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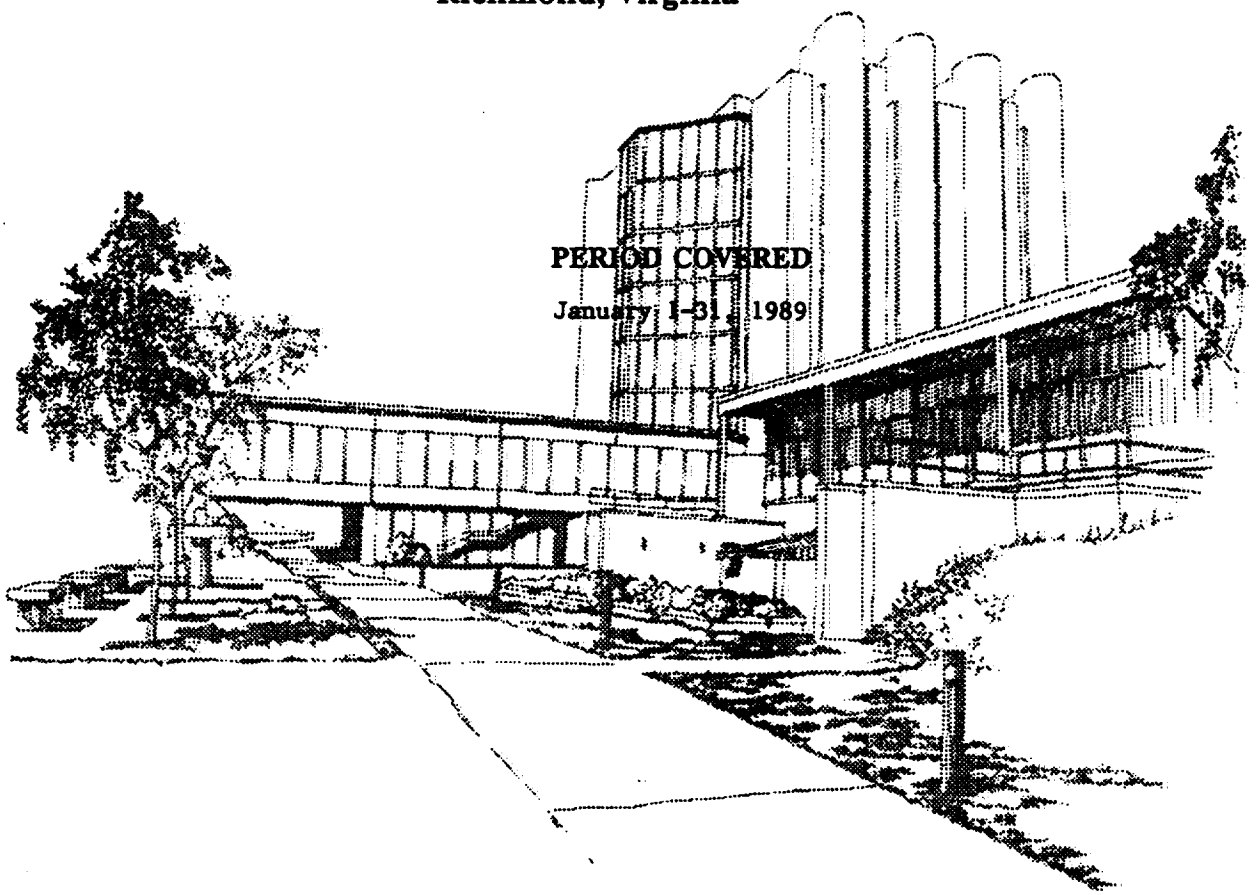

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January 1-31, 1989

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PROJECT NUMBER: 2107
PROJECT TITLE: Filter Research & Development
PROJECT LEADER: C. J. Campbell
PERIOD COVERED: January, 1989

I. IMPROVED FILTRATION - INCREASED EFFICIENCY:

A. **Objective**: Develop filter systems with a higher efficiency than presently available and evaluate them for subjective advantages.

B. **Status**: Cigarette models using Celanese CA web and Eastman 1.6dpf tow filters have been made in both 6mg and 2mg models as part of projects 605 and Merit Super Lights. These have been analytically smoked by CI and are now being tested by Product Development on the OC panel along with models made with paper filters.

Sample filter rods made from CA web were produced at Intertaba and were evaluated for variability. The CA web rods showed more variability than we normally experience with CA tow, but showed less variability than samples produced by Celanese from the same batch of material. Intertaba may have a processing advantage over Celanese. This will continue to be pursued.

Engineering examined the web fed rodmaker at Intertaba and obtained needed information. This machine will now be shipped to Richmond for our development work.

II. IMPROVED FILTRATION - MENTHOL STABILITY:

A. **Objective**: Investigate methods of improving the stability of menthol delivery in smoke of aged cigarettes.

B. **Status**: Sample filter rods made with citric acid and potassium citrate added to PVA menthol were produced and evaluated on Marlboro Lights rods. Menthol in smoke was far below target.

III. IMPROVED FILTRATION - NOVEL FILTER SYSTEMS:

A. **Objective**: Develop and evaluate new and unique cigarette filters which may offer a distinct product advantage.

B. **Status**: CONCENTRIC FILTERS: Filtrona is in the process of making peripheral flow concentric filters with CA web in the core for our evaluation. The high efficiency of the CA web will filter out more of the core smoke and might have a subjective benefit.

CARBON FILTER ROD: Seven new samples of extruded carbon filter rods made from Lark Milds carbon were received from Stackpole on 1/11/89 and on 1/17/89. Two of those samples were too brittle to be useable. Test cigarettes have been made and submitted to CI for gas phase analysis. The carbon rods were not crushed, since there was sufficient smoke flow through the three star shaped

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holes. Several carbon rods were placed on a combiner at Intertaba for a brief test. The rods were difficult to cut and resulted in damage to the combiner knife.

IV. **IMPROVED FILTRATION - SELECTIVE FILTRATION**:

A. **Objective**: Explore the use of specific additives in filters for selective filtration or subjective modification of smoke.

B. **Status**: FML: Analytical smoking evaluation of full flavor cigarette models containing an acidic additive and various polar hardeners on FML filters is still in progress. Preliminary data indicates that treated FML is an improved filter for the smoke substances that are delivered by a normal FML filter at higher levels than by a CA filter. The subjective screening of these models by Flavor Development personnel is in progress and should be complete by the end of January.

Plans are to proceed with a second set of models with other types of additives and hardeners, other than carbowax, as the carrier.

NICOTINE REDUCTION: Product Development has remade models of GCC Marlboro LS cigarettes with filters containing 4% ethyl citrate/ 4% triacetin (9% nicotine/tar reduction), and is performing a large scale subjective evaluation.

V. **MODEL FILTER PERFORMANCE**:

A. **Objective**: Put the filter modeling program into practical use.

B. **Status**: The filter model on the Sun Workstation is becoming a useful design tool and has been used to design low delivery cigarette models for Project 605 and Merit Super Lights.

VI. **FILTER SUPPORT FOR MAJOR PROGRAMS**:

A. **Objective**: Provide design assistance and potential new filter systems for major R&D programs.

B. **Status**: LOW SIDESTREAM: Cigarette models at both 17mm and 24mm circumference with a carbon on tow dual filter were made and subjectively evaluated. Filter rods with lower carbon loading are now being made and will be evaluated. The Semiworks has been requested to obtain parts and prepare a combiner for the 24.0 filters. Currently, this combining is being done at Stockton. A meeting is planned with representatives from Filtrona to discuss potential test market quantities of 17mm carbon filters.

ART: PVA menthol filters have been made and will be attached to ART cigarettes in an effort to boost the menthol delivery to the desired level. Previous ART cigarettes mentholated with maximum menthol obtainable on foil did not deliver sufficient menthol.

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PROJECT NUMBER: 2108
PROJECT TITLE: New Product Technology
PROJECT LEADER: W. T. Callaham
PERIOD COVERED: January, 1989

I. SAUNA

A. **Objective**: Develop an acceptable product with a modified plastic fluted filter.

B. **Status**: Prototypes with cork-on-white tipping (Merit-type) were made with a single dilution level and two different flavor systems. The cigarettes were diluted as follows: 12% front-band, 80% rear-band, and 130 Coresta paper. ISO smoking results are as follows:

	Sauna Flav 1		Sauna Flav 2		Saudi Barclay	
	Open	Occl.	Open	Occl.	Open	Occl.
Tar, mg/cigt	1.8	10.9	2.2	11.3	1.5	11.8
Nicotine, mg/cigt	0.18	0.78	0.23	0.81	0.18	0.96
Puffs/cigt	8.2	6.2	7.8	6.4	9.8	8.0

Smoke blowback continues to be less than that of Saudi Barclay. Furthermore, internal and EEMA subjective evaluation indicates a preference for Sauna over Saudi Barclay. The flavor system of choice is the Congress flavor (flavor 2).

A shipment of 1800 Sauna cigarettes were shipped to FTR for evaluation of a new smoking machine mouthpiece which simulates human lip occlusion. ISO testing of the Sauna and the Saudi Barclay at FTR was requested for comparison to our results.

An ESP perforator was transferred from the DMCP Facility to the filter development lab to support further paper perforation development.

C. **Plans**: A final set of Sauna models will be produced in February to adjust the burn additive level and the puff count. Paper citrate level will be reduced from 2% to 0.6%.

After determining the final design specification for the regular Sauna model, the maker will be modified to begin development on a dual charcoal/fluted filter model for Japan.

II. HUMIDOR PACK

A. **Objective**: Develop a moisture release device for use in a cigarette pack which maintains the pack OV at the desired level.

B. **Status**: Materials have been prepared for a packet production trial at Paco Pharmaceuticals in mid-February.

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C. **Plans**: The packets from the Paco trial will be used if possible for a consumer test in the Saudi region in mid to late March. Positioning of the packet at the front and rear of the cigarette pack will be tested. Also, the cigarette packs will be 1.5 mm deeper to accommodate the packet and a 24.8 mm circumference cigarette.

Some of the packets from the Paco trial will be used for cigarette packer insertion trials to be conducted by PM Engineering.

III. KAYMICH MENTHOL APPLICATOR

A. **Objective**: Evaluate a Kaymich menthol applicator as an alternative mentholation process.

B. **Status**: Mentholated samples were produced to compare Kaymich mentholation versus foil mentholation and spray application using both mentholated and non-mentholated filters. These are being analyzed for menthol content, delivery and subjective impact throughout six weeks of hold time. To-date, analytical results after two weeks indicate that the spray applied models deliver more menthol in smoke than either the Kaymich or the MOF models at comparable filler and filter menthol loadings.

Internal subjective testing through three weeks indicates that the Kaymich models are subjectively comparable to both MOF and spray applied models, both with and without mentholated filters.

Kaymich variability of menthol application to the paper was evaluated via high-speed video and appeared to be uniform. Rod-to-rod menthol content is also being evaluated.

C. **Plans**: A consumer test of Kaymich models is being considered for late-February to early-March with 100 mm Marlboro Lights Menthol SP. Therefore, the Kaymich unit will be relocated to a maker currently setup for 100 mm cigarettes. The next trial on this maker will also test an in-line nozzle that applies menthol to the filler instead of the paper. The second Kaymich unit will be installed on the Rothmans mentholator in mid-February to apply molten menthol to foil.

IV. EMBOSSING TECHNOLOGY

A. **Objective**: Explore embossing technology for potential new product development.

B. **Status**: Dual cigarette papers were prepared for Project Trim with several different calendering patterns. These included full face calendering, single and double-edge calendering, and full-face verge-line calendering. Acceptability is being based upon overall cigarette performance and aesthetics. The challenges with calendering are the resultant reduction in paper porosity, tendency to smear verge lines and inner wrap wrinkling occurring

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at the maker. To-date, the most promising paper appears to be one with hard single edge calendering.

Also, several variations of cigarette paper calendered in a banded pattern were prepared for Project Tomorrow. These are being used in a study to determine the conditions at which the cigarette does not sustain smoulder.

C. **Plans**: A modified verge embossing roller is being fabricated to provide verge lines which do not smear upon calendering. This will be available in early February. Also, a design is being drafted for slitting capability on the embossing unit.

Continue providing support as needed and pursue other applications for new product development.

V. MENTHOL-ON-FOIL

A. **Objective**: Support the introduction of the menthol-on-foil process.

B. **Status**: Mentholated foils were prepared for Product Development as requested.

C. **Plans**: In conjunction with the Kaymich program, evaluate molten menthol application to foil at the Rothmans mentholator.

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PROJECT NUMBER: 2304
PROJECT TITLE: Flavor Development - New Products
PROJECT LEADER: Barbara G. Taylor
PERIOD COVERED: January, 1989

I. LOW DENSITY ROD

A. Objective: To achieve unique products via the Low Density process.

B. Status: The Low Density Rod Design study is under way. The following blends have been produced and awaiting cigarette manufacture:

- 400 lb. LDB-2R blend with Half Pint Casings and A/C
- 400 lb. High ET Experimental blend with increased Half Pint flavors
- 400 lb. LDR Experimental blend.

The Aftercut Study models with 1x, 1.5x and 2x levels of Marlboro aftercut are awaiting analysis of the anethole levels.

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PROJECT NUMBER: 2305
PROJECT TITLE: Flavor Development-Brand Modification-Menthol/Distinctive
PROJECT LEADER: H. M. Maxwell
PERIOD COVERED: January, 1989

I. ALTERNATIVE FLAVORS:

A. Objective: To identify and qualify alternative sources for flavoring materials.

B. Status: Results of each replicate POL testing of alternative Jonex indicate no difference for each test. Specifications are being determined.

C. Plans:

Recommend Qualification of the Alternative Jonex	January, 1989

II. PROJECT MOOG:

A. Objective: To develop the expertise to produce cigarettes that are subjectively equivalent to Salem, Newport and Kool.

B. Status: A K-type POL test was remade in Semi-works. Cigarettes are being overtipped. A POL is being requested for the S-type model.

C. Plans:

K-type POL Shipment	January, 1989

III. MENTHOL RELEASE COMPOUND:

A. Objective: To develop a mentholated charcoal filtered cigarette utilizing a menthol release compound. Also, to apply menthol release technology to other areas.

B. Status: Cigarette production utilizing MGC application via direct chimney application is scheduled in Semi-works. MGC and Polymic are being considered in other programs.

C. Plans:

Cigarette Remake (MGC)	January, 1989

IV. ALTERNATE HUMECTANT PROGRAM:

A. Objective: To develop the capability to produce cigarettes that are propylene glycol and glycerin free, while maintaining product subjective integrity.

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B. **Status**: Cigarettes with casing modifications were made in Semi-works with modified flavor systems designed to improve subjective characteristics of PG/G-free cigarettes. Subjective evaluations of 100% PG/G-free ES and DIET cigarettes are in progress to requalify those materials for testing.

C. **Plans**:

PG/G-free POL	April, 1989

V. **PROJECT LEVO**:

A. **Objective**: To determine the subjective differences between natural and synthetic menthol, and identify techniques to lessen PM dependence on natural menthol.

B. **Status**: Flavor development work, utilizing menthol isomers and analogous compounds, is in progress. Another POL testing 100% synthetic versus 100% natural menthol is being requested.

VI. **LOW TAR CIGARETTE DEVELOPMENT**:

A. **Objective**: To improve subjective attributes of ultra-low tar cigarettes.

B. **Status**: Testing of models is in progress on the North Complex Panel. Additional models were made to slightly reduce puff count. Analyses are in progress. This program is being merged into the Merit Super Lights program. Models will be made incorporating flavors developed for that program.

C. **Plans**:

Evaluate Cigarettes with Reduced Puff Count	January, 1989
Flavor Optimization	Ongoing

VII. **PROJECT GRAIN**:

A. **Objective**: To significantly reduce alcohol levels in PM flavor systems, while maintaining product subjective integrity.

B. **Status**: Aftercut flavors were processed at Twentieth Street without appreciable anethole lose. Control and test cigarettes, with treated aftercut, were made in Semi-works. Analytical and subjective evaluations are in progress.

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PROJECT NUMBER: 2306
PROJECT TITLE: Marlboro Standardization and International Support
PROJECT LEADER: J. L. Spruill
PERIOD COVERED: January, 1989

I. MARLBORO STANDARDIZATION

A. **Objective**: Analytical and subjective evaluations of production Marlboro KS/LS.

B. **Status**: Analytical and subjective evaluations of Standard Run VI complete. POL's 3610-3614 were cancelled due to variability of the Winston product. December 21, 1988 factory pick-up under evaluation.

C. **Plans**: Analytical and subjective completion of December 21, 1988 factory pick-up.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL

A. **Objective**: To provide subjective direction for programs within R&D and manufacturing locations.

B. **Status**: Eighteen panels completed for the reporting period in addition to three brand profiles and forty menthol evaluations for the Flavor Development Menthol Aging Study.

C. **Plans**: Provide assistance as needed.

III. PROJECT NATURAL

A. **Objective**: To develop 85mm and 100mm full-flavored and lights prototypes using blend components and flavor systems which will result in a natural blended product.

B. **Status**: POL 3598 (Blend C using limited flavor ingredients vs. Marlboro 85) was remade the week of January 16, 1989. Lights prototypes will be made from this filler for internal evaluation.

C. **Plans**: Analytical and subjective evaluations of POL 3598.

IV. FLAVOR RESOURCE DATA BANK

A. **Objective**: Creation, customization and maintenance of flavor resource and data files for use by Flavor Development Division.

B. **Status**: New vendor samples continue to be evaluated subjectively.

C. **Plans**: Division updated with information.

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V. INTERNATIONAL SUPPORT

International Brands Smoking Panel

A. **Objective**: Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.

B. **Status**: Sixteen panels completed during the reporting period as well as evaluations of L&M Milds FTB production start-up.

C. **Plans**: Provide assistance as needed.

PROJECT MOUNT ULTRA-JAPAN

A. **Objective**: Explore the use of new blends, new flavor systems and different construction styles. Cigarettes will be in the low and ultra-low category for the Japanese market.

B. **Status**: Awaiting blend development for flavor system evaluations.

PROJECT OLYMPIC (KOREA)

A. **Objective**: Development of a product to be competitive to Pine Tree King Size.

B. **Status**: Casing and aftercut systems still in progress.

C. **Plans**: Prototype making will begin shortly with a 1500-pound run in Semi-works.

MARLBORO IMPROVEMENT PROGRAM (AUSTRALIA)

A. **Objective**: Develop an Australian Marlboro subjectively closer to the U.S. Marlboro.

B. **Status**: Prototypes did not meet subjective objective. Further blend development is planned.

C. **Plans**: Blend reformulation.

MEXICO

A. **Objective**: Modifications of the existing Marlboro flavor system to be brought in line with U.S.

B. **Status**: On-site involvement is underway to make necessary flavor modifications.

C. **Plans**: On-site testing of product modifications.

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PROJECT NUMBER: 2307
PROJECT TITLE: Flavor Investigation/Nonvolatile Flavor Investigation/Processed Tobacco
PROJECT LEADER: W. R. Raymond
PERIOD COVERED: January, 1989

I. FLAVOR INVESTIGATION:

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Results:

1. Analytical Support: Flavor and casing analyses have been completed for several factory trials including Merit Lts., Japan, POL 3598, Marlboro Std. VI, Famous and Half-Pint. Concentrate, filler and A/C analyses for anethole and vanillin were completed for Europe Marlboro.

2. Project ART: Flavor analytical specifications were completed for ART flavor formulae transmittals. A revised cooked flavor for ART casing was developed, analyzed and subjectively compared to the original flavor with promising results. All packaging materials for ART Ad-Pack were analyzed for residual solvent content by headspace GC. All were released for semi-works production.

3. Low Density Rod: GC analyses were completed of anethole in filler distillate/extracts for an LDR flavor survival study.

4. Semi-Works A/C Optimization: Five A/C samples and 60 filler samples were analyzed for anethole for Phase 2 of an A/C application optimization study in Semi-works. The study is complete and a report is expected shortly.

5. TRIM: GC analysis continued of three TRIM A/C's (6 month old) to determine storage stability.

6. Work continued to adapt for Flavor Development use a GC-TCD method for determination of water, ethanol, PG, and glycerin in casings and A/C's.

II. PROCESSED TOBACCO:

A. Objective: To develop basic and applied knowledge for the purpose of improvement or selective modification of subjective properties of processed tobaccos.

B. Results:

1. ART Stem Utilization: Results were returned from POL 3609, testing post-ART stem substitution for 1/6 of Bright stem in RL's (Marlboro Blend). Panelists found significant

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differences between the test and control Marlboro. Marlboro smokers preferred the control, rating it higher on mild taste, good taste and aftertaste while Winston smokers rated the test higher on fast burn and dry taste. Since more recent pilot RL's with 1/3 and 1/6 levels of post-ART stem have shown similar subjective results, lower utilization levels will be tested.

2. Dry Flavor Replacement: Recent native roasted JONEX submissions from two alternate vendors have moved closer to each other and to the original vendor material in composition. Solid extracts have been prepared and analyzed and will be incorporated in pilot RL sheet for subjective comparison to the original material.

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PROJECT NUMBER: 4009
PROJECT TITLE: Smoke Studies
PROJECT LEADER: B. L. Goodman
WRITTEN BY: B. L. Floyd
PERIOD COVERED: January, 1989

I. TRIM V

A. **Objective**: Develop subjectively acceptable 17 mm circumference cigarettes with reduced sidestream visibility.

B. **Status**: Various models of the 100 mm, 17 mm circumference cigarettes were made in single and doublewrap configuration using the special $CaCO_3$ paper with CMC and MAP from Kimberly-Clark. The inner liner being used is a low-chalk paper from DeMauduit, and the outer wrap is electrostatically perforated to 60 Coresta. Cigarettes were made at Semiworks using Trim IV filler with no aftercut and with an aftercut chosen for further development. Several models also included filters with two different levels of charcoal loading. The doublewrap models continue to result in significant reductions in sidestream smoke.

Development work continues on potential ways to laminate the two layers of paper together. Kimberly-Clark supplied samples of paper in which the outer paper had been laminated to a very thin "tea-bag" paper using two different levels of CMC. The increased level of CMC caused this paper to be subjectively unacceptable. Other efforts of paper lamination include various methods of calendering the papers together. One of these involves a "verge-line" calendering pattern using an outerwrap paper with no verge lines, and the calendering gives the finished double paper the appearance of standard cigarette paper containing verge lines. Initial machinability testing of this paper was successful. Another, more promising, method involves calendering the two paper together along their common edge.

II. Lotus

A. **Objective**: Develop subjectively acceptable 24 mm circumference cigarettes with reduced sidestream visibility.

B. **Status**: Additional development work has begun on low sidestream cigarettes in a 100 mm, 24.0 mm circumference configuration using 35% $Mg(OH)_2$ paper. Initial models were made with 100% expanded material, high-expanded blend, and the low sidestream blend. The low sidestream blend was found to be the most acceptable subjectively. Additional models have been made with the low sidestream blend and several doublewrap configuration. Analytical results are pending.

Charcoal filters with additional, varying levels of charcoal loading will be tested to determine optimum level for subjective improvement. Combinations of doublewrap papers will continue to

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be tested to achieve the most desirable analytical and subjective results in a finished cigarette.

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PROJECT NUMBER: 2304, 2305, 4015
PROJECT TITLE: New Product Development
PROJECT LEADER: B. G. Taylor, H. Maxwell, J. B. Easley, and L. S. Wu
PERIOD COVERED: January, 1989

I. MARLBORO ULTRA LIGHTS

A. **Objective**: To develop 85/100 mm Ultra Low (6 mg) candidates for Marlboro line extensions.

B. **Status**: Specifications are being written. Test market scheduled for May, 1989, for box, king size and 100mm versions.

Factory trial in Cabarrus scheduled for the week of 2/13/89.

C. **Plans**:

Factory Trial (Cabarrus)	Feb. 89
Test Market	2nd Qtr.'89

II. Trim I

A. **Objective**: To develop ultraslim product candidates with 17mm circumference that demonstrate product advantages over Capri and Capri Menthol.

B. **Status**: POL 7182 (Model 2 Regular vs. Capri) and POL 9088 (Model 2 Menthol vs. Capri Menthol) cigarettes will be produced after the Trim V Ad/Pack. These cigarettes will have to be hand packed.

C. **Plans**:

POL Shipment	March '89
Test Market	TBD

III. Trim V

A. **Objectives**: To develop ultraslim product candidates with 17mm circumference with reduced visible sidestream that demonstrate product advantages over Capri and Capri Menthol.

B. **Status**: The model selected for the Trim V Ad/Pack is the Trim IV blend with Marlboro casings and aftercut 8544-49, perforated paper 2123-83 as outer wrap and DeMauduit's 535-17c as innerliner. This combination gives 70-75% sidestream visibility reduction. Cigarettes for the Ad/Pack will probably be made on the baby Hauni in Semi-works. Cigarette production is scheduled for the week of February 13.

Work continues on investigating charcoal/CA filters as well as an all natural casing.

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C. Plans:

Combined Filter Prototypes	Feb. '89
Ad/Pack	1st Qtr. 89
Paper Evaluations	On-going
Test Market	July 1989

IV. PROJECT NATURAL

A. Objective:

Phase I - To develop "All Natural" cigarette candidates with no additives to the tobacco.

B. Status: Flavored POL 3598 (97K Flavor system vs. Marlboro) was remade week of 1/23/89 and awaiting subjective and analytical information.

C. Plans:

Study Anti-Spotting Papers	On-going
Menthol	On-going

V. PROJECT EXTRA

A. Objective: The development of 4-8 mg product candidates that have the taste of products with twice the tar.

B. Status: Work continues on investigating vapor/particular split of low density cigarettes. In early January, 57mm and 88mm rods were made at 40% weight reduction, 25% weight reduction and regular density, with 19 sec and 35 sec cigarette papers. Tubes have been hand attached for puff-by-puff unfiltered smoking at three puff volumes (20cc, 35cc and 45cc). The cigarettes have been submitted to CI.

Cigarette designs have been completed for a study of low density vs. expanded at the 6mg and 2mg tar ranges. The control (standard blend and high ET blends) cigarettes have been made and are in CI. The low density cigarettes will be made in early February.

The Low Density Rod Aftercut Study models with 1x, 1.5x and 2x levels of Marlboro aftercut are awaiting analysis of the anethole levels.

C. Plans:

Evaluate alternate filters	On-going
Puff profile	On-going
Low density rod program	On-going

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

VI. Project 605

A. Objective: To develop a 6mg free-standing cigarette which appeals to flavor low smokers.

B. Status: Blend A (32% partially expanded bright and burley) with aftercut 120-C was selected as the best model with both CA and paper/CA filters. Cigarettes with both filter configurations are being evaluated on the OC Panel. POL 0212 (nonmenthol monadic) has been made and released for shipment. This POL contains the CA Filter model.

C. Plans:

POL Testing	1st Qtr. 89
Filter Evaluations	On-going

VII. Merit Super Lights

A. Objectives: To develop a 2mg "Merit-type" cigarette.

B. Status: A POL has been requested for a new model (Model 2) that incorporates the 350°F expanded burley blend, 7695-163D total blend casing and 7695-178E aftercut vs. the 25% BLDET model. Model 2, the 25% BLDET model and PM Ultra Lights were smoked on the Internal OC panel. The results indicated the 25% BLDET model was lower in strength than the other cigarettes.

C. Plans:

POL test Production	1st Qtr. '89
Prototype Evaluations	Ongoing

VIII. PROJECT VALUE ENTRY

A. Objective: To develop products to compete in the Value Entry Categories.

B. Status:

Alpine

National introduction was January 2, 1989 for all four packings of KS and 100"s. Long Size FTB packings introduction was January 2, 1989 in New York only.

Target

POL 3608 (Target Model II 85mm vs Winston 85mm) was requested to determine the consumer acceptability of the new Target blend and flavor system versus Winston by Winston and other RJR smokers. The test was shipped January 5, 1989 and the closeout date was February 1, 1989. Currently awaiting test results.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

C. **Plans**:

Alpine National Introduction	1/89
Target Ad/Pack Testing/Factory Trial	On-hold

XI. Marlboro Blend Reformulation

A. **Objectives**: To remove off shore tobacco from Marlboro blend.

B. **Status**: POL testing (3605 and 3607) of blends XI and XII released for shipment. Results available mid-March, 1989. These blends will also be tested on the Marlboro Monadic Panel.

C. **Plans**:

POL testing	1st Qtr. 89

X. Cartier

A. **Objectives**: To develop a Cartier similar to Europe's product for the U.S. market.

B. **Status**: The All Natural blend "C" initial prototypes have been produced.

C. **Plans**:

Prototype Production/Evaluation	1st Qtr. 89
Consumer Testing	TBD
Test Market	July, 1989

IX. Lotus

A. **Objectives**: To develop a 24.0 circumference 100mm low sidestream product with 70% reduction in visibility to compete against Vantage Excel.

B. **Status**: A conventional type blend (2A) and a highly expanded blend (82% XTH/18% MT Oriental) are currently undergoing evaluations with $Mg(OH)_2$ paper and charcoal filters. Dual wrapped systems will be investigated for possible subjective improvement. The 2A blend configuration is currently the preferred model.

C. **Plans**:

Prototype Production/Evaluation	1st Qtr. 89
Consumer Testing	2nd Qtr. 89
Test Market	TBD

2022172042

Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 4022
PROJECT TITLE: International Product Development
PERIOD COVERED: January, 1988
WRITTEN BY: A. H. Confer

I. V-88 PROJECT (PANAMA)

A. **Objective**: Develop a product for Panama to compete against the local Viceroy.

B. **Results**: Prototypes with a new blend have been produced and submitted for analytical and subjective evaluations.

C. **Plans**: Subjective results to date indicate that additional casings and/or flavor systems will need to be evaluated.

II. PROJECT DRAGON (PEOPLES REPUBLIC OF CHINA)

A. **Objective**: Develop a "blended" KS cigarette to be a non-PM trademark owned and manufactured by CNTC. Tar targets: 17-19 mg (GCF-2 factory), and less than 15 mg (ZCF factory).

B. **Results**: Analytical results from GCF-2 prototypes made at GCF-2 and smoked in Richmond and China demonstrated comparable numbers except for a 4mg descrepancy in FTC tar deliveries.

C. **Plans**: Contacted Hong Kong office for comminucation with GCF-2 pertaining to analytical results.

III. MARLBORO IMPROVEMENT (MEXICO)

A. **Objective**: Evaluate the Mexican Marlboro made with USA produced flavor concentrates.

B. **Results**: Marlboro LS control and Marlboro test #1 (using USA flavor concentrates) were prepared the week of January 15th.

C. **Plans**: Samples have been submitted for analytical and subjective evaluations. Results are expected the first of February. A consumer test is scheduled in Mexico for February 1, 1989.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

IV. MARLBORO CLASSIC (AUSTRALIA)

A. Objective: Develop Marlbor 83mm Box with a 13.5mg tar and Marlboro Lights Box (regular & menthol) at 10.5mg tar.

B. Results: Prototype with the Marlboro USA blend was selected.

C. Plans: Proceed with the development of the Marlboro Lights regular and menthol products.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 5001
PROJECT TITLE: Packaging Studies
PROJECT LEADER: H. R. Dunaway
PERIOD COVERED: January, 1989

PACKAGING STUDIES

A. **Objective**: Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. **Status/Plans**:

Residual Printing Solvents in Packaging Materials: Coordinated evaluation of the following items:

- Printkote Hi-Gloss FT Board Stock
- Pre-applied carton insert adhesives:
  Craigbond 3195
  Craigseal 14951
  Craigbond 3710F
- Genco Filter Lts Men SP 20
- Genco FF KS SP 20
- Genco Filter LTS SP
- Genco Ultra Lts SP 20
- Genco Non-Filter SP 20
- CJW1654 & CJW1655 Water Base Lacquers
- Alpine 100 SP 20 Two versions of 2L-978A
- Clairex Aqua-Coat GC-563 (Stone Container)
- Offset Printed, Promotional Corrugated Mailer Label
- B&H DUL Men FT 20 W/Canadian and Domestic Inks
- Offset and Gravure printed materials from Japan Tobacco, Inc.
- Zonyl FSN Surfacant (Thiele-Engdahl)

Poly Laminated Blanks and Cartons: Week 4 sampling of 12 week OV study was completed.

Fiber-Lam, Inc. Shipping Cases: Twelve week storage study was completed. Report in progress.

Miscellaneous: After six weeks storage under adverse environmental conditions, Hostaphan RN36D Heat-Sealable Tear Tape proved to be comparable to currently used materials in appearance and function. This material is being evaluated for use on new generation high speed packaging equipment.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 0400
PROJECT TITLE: Low Density Rod Development
PROJECT LEADER: R. S. Mullins
PERIOD COVERED: January 1989

I. LOW DENSITY ROD

A. **Objective**: Develop a continuous process for the production of reduced density cigarettes.

B. **Results**: Firmness results from the filler OV test indicated that cigarette firmness improves with increasing filler OV over the range tested (12% to 16%). The equilibrated firmness also appeared to be independent of the amount of water removed from the filler during the drying step.

A test was conducted to determine if the addition of moisture to the filler prior to steaming improves bonding of the rod. An ultrasonic spray nozzle located underneath the suction tape just downstream of the ecreteur was used to increase the filler OV by either 2 or 4%. The moisture application did not appear to affect operation of the maker during the test. Visually, the rods made with the added moisture appeared better bound. Test results indicated that the cigarettes produced with the 4% moisture addition were 0.2 mm more firm than the control cigarettes.

Production of low density rods for a study being coordinated by Duane Watson and Cathy Ellis was completed. This study will compare the smoke properties of conventional, normal density cigarettes to those of reduced density cigarettes produced via either the bonding process or the expansion process.

No problems were encountered in producing low density rods from the all-lamina ripper shorts generated from the ripper test at Stockton Street. However, at a 25% density reduction, the quality of the rod was somewhat lower than normal. No additional binder had been applied to the filler after the ripping test.

A study is underway to determine if the vapor to particulate TPM ratio of cigarettes having a 40% density reduction is significantly different from that of normal density cigarettes. The vapor to particulate ratios will be determined by analyzing the puff X puff deliveries of the cigarettes. Testing of the cigarettes is in progress.

The analytical data from the Project Tomorrow samples has been received. Three models, varying in aftercut level, have been submitted for subjective screening to identify the one to be submitted to the MC panel.

Testing and tuning of the automatic weight control system on the maker is underway.

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C. **Plans**: Produce samples for a test comparing the subjective and analytical properties of low delivery cigarettes produced using the low density process to that of cigarettes produced conventionally using high DIET blends. Design and install a system to apply moisture to the tobacco bed on the suction tape just downstream of the ecreteur which is suitable for routine sample production. Complete testing and tuning of the automatic weight control system.

II. KAYMICH MENTHOL APPLICATOR

A. **Objective**: Evaluate the Kaymich direct menthol applicator as an alternative mentholation process.

B. **Results**: The Kaymich unit in Semiworks is currently being moved to a 100 mm maker at the request of New Products.

Production of samples for the menthol aging study comparing the Kaymich process, menthol-on-foil, and the standard aftercut process was completed. Testing of the samples is being coordinated by New Products.

Plans have been made to evaluate the use of the Kaymich unit to apply molten menthol to foil as an alternative to the technique used in the current menthol-on-foil process which requires that the menthol be applied in an ethanol solution. Development Engineering is designing a new nozzle for the Kaymich unit suitable for use with the Rothmans foil coater in Semiworks. The design and fabrication of the nozzle and a mounting bracket is scheduled to be completed by February 10. The preliminary trials to determine the feasibility of this approach will be conducted during the week of February 13.

A test program to evaluate the long term stability of the Kaymich system's delivery rate and its sensitivity to variation in various process variables has been developed. This work will be carried out using the same Kaymich unit as the foil application trial and will therefore not be undertaken until the preliminary testing of the foil application concept is completed.

C. **Plans**: Determine the feasibility of applying molten menthol to foil using the Kaymich applicator. Initiate studies to determine the sensitivity of the Kaymich unit to variations in process variables. Assist New Products in producing 100 mm product samples. Determine if the maker side rail modified for use with the tobacco stream application nozzles can be used with the 100 mm maker. If so, install and evaluate the nozzles designed to apply menthol directly to the tobacco stream, otherwise obtain and modify a rail for the 100 mm maker. Develop and publish plans for addressing the process development needs of this program.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 1307
PROJECT TITLE: Reconstituted Tobacco Development
PROJECT LEADER: R. G. Uhl
PERIOD COVERED: January, 1989

I. IMPROVED SHEET PROPERTIES

A. Objective: Improve the physical characteristics and blend properties of reconstituted sheet materials.

B. Results:

1. ART Project - POL results evaluating the acceptability of utilizing half of the ART stems in RL showed a preference for the control (pilot RL in both the test and control cigarettes). A second set of pilot RL sheets made with current ART stems (PG/glycerine precasing on the extracted filler) were subjectively quite comparable to the POL test sheets in blends, and represented no improvement. Full ART stem utilization pilot sheets made from both of these ART stem lots were also very similar in blends; these were more subjectively detectable than the half utilization sheets, but less so than would be expected from doubling the ART stem.

   The second lot of stems (precased ART filler) was also used to make pilot RL utilizing a quarter and an eighth of the ART stems. These will be screened in the normal TC/150B combination. Blends will also be made with individual sheets of the various utilization levels (e.g., test TC with control 150B) in order to determine if ART stem inclusion in a particular RL type is more subjectively feasible.

   RCB handsheets will also be made at lower ART stem inclusion levels (half utilization was subjectively obvious in handmade 100% cigarettes). Additional handsheets will be made using postwashed ART stems, simulating washing of the ART stems along with (or in lieu of) the burley stems at the BL Plant.

2. Humectants - Blends made with PG/glycerine-free strip casings were found unacceptable (dry, metallic), attributed to excess sugars in the strip casings (sugars used to replace PG and glycerine). The casings are being reformulated for retesting. Cigarettes were made of production PG/glycerine-free sheet materials (24% blend level) and expanded blend components (100% cigarettes) for subjective screening.

C. Plans:

1. Expedite cigarettes with low ART stem levels in RL, and test/control RL combinations.

2. Evaluate lower ART stem levels in RCB.

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3. Produce sheets from ART stems with Hauni tunnel treatment, and sheets from top and bottom absorber baskets.

4. Expedite evaluation of PG/G-free blend components and casings.

II. SUBJECTIVE MODIFICATION OF RL

A. **Objective:** Improve or modify the subjective character of RL.

B. **Results:**

1. Modified 150B - Results from RL pilot trials showed that the Branson ultrasound unit was unsuccessful in eliminating phosphate precipitate rejection from Modified 150B size at the size press nip. No further pilot work on Modified 150B is planned; pursuit of RCB subjective flavor in RL will require the creation of a new flavor system.

2. Dry Flavor Replacement - Initial large analytical differences in liquid flavor samples from the three potential vendors necessitated an iterative process wherein Flavor Development analyzed samples of native extracts (as extracted, without sugar or color adjustment) and recommended changes in roasting conditions, resulting in new samples for analysis. The latest samples from Madis and Takasago are analytically close to each other and reasonably close to the Chart "control" sample. These have passed initial screening (sprayed on unflavored RL) and larger quantities for pilot trials are enroute.

C. **Plans:**

1. Produce pilot RL-150B with individual liquid flavor samples.

III. PAPERMAKING TECHNOLOGY

A. **Objective:** Support development of proprietary cigarette papers for low sidestream and other new product applications.

B. **Results:**

1. Handsheets - The new Coresta porosity unit was installed in the handsheet lab and calibrated. Handsheets were made at a range of porosities to evaluate fillers (dolomite, magnesium carbonate, calcium carbonates of various and mixed micron sizes). Unfilled papers were made and sized with various burn rate additives and a burn rate retardant. Evaluation of sol-gel coating techniques was initiated.

   Plots of porosity vs. airflow through papers (handsheets, Maine pilot papers and commercial papers) gave a straight line relationship indicative of air flow through pores. Plots for perforated or holed papers are nonlinear, indicative of orifice flow.

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Handsheet work concentrated on simulation of Trim-5 double wrap papers prior to pilot trials at the University of Maine. These included the inside paper (18 gm, 3% carbonate), the outside paper (45 gm, 35% carbonate), a single 63 gm composite paper with prorated carbonate content (26%), and a bilayer paper made by wet pressing the 18 and 45 gm papers at couching moisture (~85% OV).

2. Pilot Trials - The above bilayer paper was produced on the Maine pilot machine by employing the secondary headbox. In commercial manufacture, this paper would be made using a second Fourdrinier wire and laminating the two sheets at couch moisture; this would better maintain the filler integrity of the individual layers and would allow separate recovery of the two white waters. Maine is also producing the corresponding composite paper,a comparable bilayer paper using wood pulp in the 45 gm layer, and a calcium carbonate/magnesium carbonate bilayer paper.

   Maine will also produce a control paper duplicating standard commercial paper for a subjective comparison test (qualification of Maine pilot papers). Maine installed the new vacuum pickup device, which has improved runnability. A second small stock screen for the secondary headbox is no longer commercially available; Maine has received construction drawings and permission to duplicate the existing pilot screen from the vendor. Maine will also try to groove an existing press roll with vergeure lines. The physical properties of European cigarette papers containing wood pulps were reviewed, and Maine was provided samples of three domestic commercial cigarette papers for physical evaluation.

   A gate roll type coater was identified as the best means of applying sizing agents, including sol-gels, with a range of viscosities. Maine will conduct preliminary trials using their new gate roll coater to determine feasibility.

C. Plans:

1. Complete Maine production of simulated double wrap papers (bilayer and composite) each at three porosities, bilayer paper with wood pulp, and bilayer Ca/Mg carbonate paper.

2. Duplicate PM commercial cigarette paper at Maine for subjective qualification of Maine pilot papers.

3. Develop rapid drying capability for the Product Development coater to prevent size migration between paper layers.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 1333
PROJECT TITLE: Semiworks Process Control
PROJECT LEADER: D. A. Phan
PERIOD COVERED: January, 1989

A. **Objective**: Evaluate and revise the process control and data acquisition system to improve processing performance and production quality.

B. **Results**:

Onspec Control/Data Acquisition Software (Oliver/Sims) -Installation of the Onspec control and data acquisition software on an IBM-AT compatible computer was completed and succesfully checked out. The software package, purchased from Heuristics Co., is currently used for historical data storage and trending of all important process points in the Semiworks large-scale primary. Data acquisition of process data is accomplished via the Fisher Controls Provox data highway. Daily process files are currently archived on floppy disks. Training was provided to the control room operators for basic operation such as data trending and archival.

Semiworks Primary Process Control System Upgrade (Phan) - Work is underway to examine the total Semiworks primary process control system and come up with a proposal to upgrade the system for improving system performance and maintainability. A project team is being formed that will include personnel from R&D TP&F, Computer Applications division, and PMUSA - Engineering.

Cutter Steam Cylinder Upgrade (Oliver) - Work is underway in collaboration with M. Webster of Semiworks Process Development group to replace the process steam and water piping for improving system perforamcne and maintainability. The project should be completed by April 89.

Modicon Datahighway Replacement (Oliver) - Parts have been ordered to replace the PLC-1 Modicon datahighway from the preblend area to the total blend for improving system reliability. The project should be completed by March 89.

Small Scale Primary Operation Upgrade (Phan/Oliver) - Instrument and electrical cost estimate for the project was provided to T. Skidmore for the preparation of the 650 appropriation request. Meeting was held with Boothe and Skidmore, to review the dryer and conditioner design. Dryer design will include ductwork and damper to bypass the incoming hot air to improve temperature control during the drying cycle.

C. **Plans**: Complete process and instrument diagram selection of a control system for the small scale operation process. Complete verification work on the Computrac moisture analyzers. Continue providing electrical plant engineering support to the Semiworks and conduct routine QA functions.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 1503
PROJECT TITLE: Modified Smoking Materials
PROJECT LEADER: W. A. Nichols
PERIOD COVERED: January 1989

I. LOW DENSITY ROD - BINDER APPLICATION

A. **Objective**: Produce precoated filler via the batch or continuous process to support the current product and process development efforts on the low density rod program. Develop a method of pre-applying binders to the tobacco in a manner that can be scaled up to commercial scale.

B. **Results**: Testing is being conducted to determine design specifications for a 10:1 scale-up of the coating process. Based on processing data, FMC, the manufacturer of the existing coating reel, suggested a 6'4" x 24' cylinder for an 800 lb/hr output. Using current coating procedures, two such cylinders would be required to achieve a 6% addition of pectin. On a production basis of three shifts per day, annual output would be sufficient for a 0.5% market share. The pectin spray system for the above cylinder is being discussed with Spray Dynamics Corp. A confidentiality agreement was recently signed.

Previous testing has shown that high OV coated filler (15%) produces improved low density rods. This can be achieved in the cylinder or by maximizing drying and reordering to 15% OV. Preliminary tests at maximum drying conditions allowed output to be increased from 80 lb/hr to 120 lb/hr. Further testing is planned.

Coated filler was produced for several product and process tests.

C. **Plans**: Further experimentation will be done with higher concentration pectin solutions in the continuous coating process to permit a 6% add-on in one pass.

II. BINDER TECHNOLOGY

A. **Objective**: Investigate the mechanism of filler bonding and stiffness produced by coating. Improve commercial feasibility by examining alternate binders and processes.

B. **Results**: Dry coating of pectin over a tackified filler was tested in the lab. Glycerin and pectin were used to precoat the tobacco. Retention to the glycerin appears greater than to the pectin precoat after a 30-minute vibration test. One of the batch dryers is being modified to dry coat 30-pound samples for cigarette fabrication.

Sample cigarettes were supplied to Mr. S. Ganeriwala for compression measurement at a range of OVs. Acoustical detection of filler breakage and debonding will also be attempted.

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A rapid pectin analysis method using the Infralyzer is being evaluated. Samples were given to Dr. T. Van Auken for feasibility studies.

C. **Plans**: Cigarettes will be fabricated from filler which has been dry coated with pectin. Laboratory reactivation testing will be conducted to determine if the 6% pectin coating is being fully reactivated during cigarette fabrication.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 1806
PROJECT TITLE: New Tobacco Processes
SECTION LEADER: S. R. Wagoner
PERIOD COVERED: January, 1989

I. PROJECT ART - PILOT PLANT SUPPORT

A. **Objective**: To provide processes for converting and casing stem materials for the Bermuda Hundred Pilot Plant.

B. **Results**: Batches of Louisville CRS (cut at 150 cpi) were cased with monopotassium citrate and shipped to the Bermuda Hundred Pilot Plant on a daily basis.

C. **Plans**: Continue to produce stem products as required by Project ART.

II. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

A. **Objective**: To conduct trials providing information for development of the ART commercial process.

B. **Results**: Process specifications for the expansion of ART filler and for the processing of ART stems were outlined for inclusion into the R&D specification package. The expansion parameters that were run in the D Pilot Plant trials were: tower temperature of 500F, tobacco throughput of 187.5 lb/hr, and tower velocity of 85 ft/sec. Due to the increased scale of the MC processing equipment, parameters will have to be modified to produce an equivalent subjective product in the MC DIET system. Where possible, the stem processing specifications were designated as those normally run by the Louisville Stem Processing Facility. Also, the monopotassium citrate casing parameters were specified.

Cigarette inspection for critical spots continued on cigarette filler by Quality Audit and Quality Engineering. One model tested was a finished cigarette in which the unexpanded portion of the blend had been processed through the Hauni tunnel, but the 20% expanded material had not. QA testing showed 10 critical spots/2000 cig. This can be compared to past results showing 2-3 critical spots/2000 cig for samples containing only filler that had been processed through the tunnel and to test results of cigarettes containing 100% expanded ART filler (non-tunnel processed) which yielded 56-76 critical spots/2000 cig. Testing was then conducted on a series of cigarettes containing feedstock filler for expansion from various processing points: exit extraction (air dried), exit the Hauni HT tunnel (air dried), and after expansion. QA inspection yielded critical spots/2000 cig values of 127, 43, and 1, respectively. Thus, to minimize spotting, ART filler for expansion must first be processed through the Hauni tunnel. Also, the Hauni tunnel must be coupled with the proper drying condition to significantly reduce spots, as evidenced by the tunnel treated, but air dried sample, which had a

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

relatively high spotting level. QE inspections (off-line device) showed the same trends.

Expansion runs of ART filler were conducted for Product Development work and Ad Pack testing. Due to downtime at the Bermuda Hundred Pilot Plant, feedstock for the previously scheduled expansion trial at the MC could not be produced. Thus, the trial will be rescheduled when run time at the BHPP becomes available.

Evaluation of the Infrared Engineering monitor for moisture, nicotine, and sugars was carried out in the laboratory by the Analytical Research Division. Analysis of data showed that the monitor agreed well with analytical OV and nicotine analyses. However, the sugar output did not correlate with analytical total reducing sugar data. The unit has been purchased, and will be further evaluated in the Pilot Plant. The development of an ammonia measurement is one potential application that will be addressed.

C. **Plans**: Complete investigation of the MC DIET system to assure that ART filler can be expanded without contamination.

Conduct laboratory studies to determine the optimum method of preparing the pre-ART filler casing solution of ammonium bicarbonate/PG/glycerin from a commercial processing standpoint.

Investigate the use of the Hauni HT tunnel to reduced the spotting tendencies of existing brands.

III. BINDER DEVELOPMENT

A. **Objective**: Develop methods to produce binder systems for the foam bonded ends and low density rod programs.

B. **Results**: In addition to preparing the standard samples of NaCMC/licorice for foam bonded ends testing by Engineering, trials were conducted with the foamer to test binder samples of varying corn syrup/licorice formulations. None performed in a satisfactory manner. Currently, polydextrose/licorice binder samples appear promising in the laboratory, and will be tested on the foamer.

C. **Plans**: Continue to provide support as required to the above programs.

IV. TMCI-ASTA SHEET

A. **Objective**: To develop a subjectively and physically acceptable reconstituted tobacco sheet using the TMCI process and PM-RCB technology for international application.

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B. **Results**: Trials were run in Tarragona to evaluate ASTA product made from tobacco ground finer than the standard grind of 60 mesh. The three tobacco grinds evaluated (95% <120 mesh, <200 mesh, and <400 mesh) all produced sheet with a satisfactory appearance. Many equipment problems were encountered which prevented the sheet being made at design specifications. The sheet products using 120 and 200 mesh dust were made from slurries with a solids content of 17.2% (design 21%). Efforts to increase the tobacco flow to the slurry mixer were unsuccessful. All sheets were made at a lower line speed (30% reduction) than the design specification. This was necessary because of the need to reduce the steam flow to the dryer to minimize condensate leaks which impair sheet quality. The casting belt was warped over about 15% of its width and condensate collection under this section impaired uniform moisture control. A list of all mechanical problems which interfered with sheet processing was given to TSA and TMCI who agreed to rectify these problems expeditiously.

C. **Plans**: Compare the survivability through cigarette making of these ASTA products made from tobacco dust <60 mesh, 95% <120 mesh, 95% <200 mesh, and 95% <400 mesh with US RCB.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 1810
PROJECT TITLE: Project ART
PROJECT LEADER: Ravi Prasad
PERIOD COVERED: January, 1989

I. PROJECT ART

A. **Objective**: To support commercial plant design and Flavor Development objectives at the Bermuda Hundred Pilot Plant.

B. **Results**: Pilot plant trials were continued in support of the Flavor Development objective. Extraction at standard conditions, i.e., 3800 psi, 140°F, 200 M/M, 3% AB/2% PG/2% G; continue to achieve the target of 97% nicotine removal and acceptable quality.

Production of DL blend filler for Ad Pack testing is in progress. After successful development work, in cooperation with the Semiworks, all Flavor Development runs are being made in the "factory simulation" mode. About 50% of the AB and humectants are applied prior to the cutters at the Semiworks. The balance of AB and humectants is applied at the Bermuda Hundred Pilot Plant. After extraction, the filler is wet cased at the Semiworks and then dried. The processing techniques for AB/PG/G have been standardized and target levels are being achieved.

In support of the commercial plant design, two prototype on-line nicotine monitors were ordered for evaluation. The first unit was received and is being installed at the pilot plant. Testing will begin in February 1989, followed by a recommendation to install in the commercial plant.

Thermal treatment of nicotine laden carbon was carried out at the pilot plant to determine scale-up parameters for commercial operation. All of the nicotine was successfully destroyed at a target temperature of 400°F. The heat-up (10 hours) and cool down (12 hours) times were long but acceptable. The furnace is being prepared for installation at the commercial facility.

A corrosion inspection program is being instituted at the pilot plant. Dye penetrant testing of all the welds on the high pressure loop has been completed. Plans are to complete 100% ultrasonic inspection of high pressure welds by the 1st quarter of 1989. Data on chloride levels at the pilot plant was provided to Engineering. An evaluation of metal corrosion potential, vis-a-vis, processing conditions and chloride levels in tobacco is in progress.

C. **Plans**: Continue production in support of Flavor Development activity.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 1101
PROJECT TITLE: Entomological Research
PROJECT LEADER: D. L. Faustini
WRITTEN BY: R. M. Lehman
PERIOD COVERED: January, 1989

I. PHYSIOLOGICAL STUDIES OF THE CIGARETTE BEETLE (CB)

A. METHOPRENE

1. **Objective**: Determine the most efficient use of the IGR, methoprene.

2. **Results**: BU treated with Kabat® sampled during processing into ET (1).

3. **Plans**:

   a. Complete studies of Kabat® protected ET.

   b. Continue bioassay evaluations of stem and scraps over time.

B. GLOBAL ECOLOGY

1. **Objective**: Relate CB ecology to the tobacco process and CB control.

2. **Results**: Pheromone trap monitoring program installed in storages and factories of several of our affiliates in Brazil to evaluate CB ecology from the farm to the cigarette (2).

3. **Plans**: Continue studies and report data collected in 1988.

C. FLAVOR EXTRACT STUDY

1. **Objective**: Determine the attractiveness of flavor extracts to the CB.

2. **Results**: Flavor extracts of fig and raisin were tested at full concentration utilizing the olfactometer and mixed-sex CBs. Both extracts were found to be unattractive to the CBs (3).

3. **Plans**: Continue evaluations of prune and apricot.

D. REFERENCES

1. Ryan, L. Memo to Young, M.D. Hogshead of Methoprene (Kabat®) Treated Burley Tobacco. December 15, 1988.

2. Ryan, L. Trip Report to Tobacos Brasileiros Ltda, a ULT subsidiary, and PM Marketing S.A. in Brazil as Part of the Global Ecology Program (in preparation).

3. Minor, M. F. Notebook No. 8539, pp. 71-72.

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PROJECT NUMBER: 1620
PROJECT TITLE: Electrophysiological Studies
PROJECT LEADER: F. P. Gullotta
WRITTEN BY: C. S. Hayes
PERIOD COVERED: January, 1989

I. NASAL EVENT-RELATED POTENTIALS (NERPs)

A. Objective: To develop methods to objectively and reliably evaluate human responses to cigarettes, smoke constituents and tobacco flavorants.

B. Results:

Cognitive NERP Study

NERP measurements are currently being obtained for the hits and misses from each of the 16 scalp recording sites for the natural vs synthetic menthol (NM/SM) discrimination. As previously reported, in the NM/SM discrimination misses more closely resembled hits than correct rejections. That is, even though subjects were failing to behaviorally discriminate between the stimuli, the NERP was coding the stimuli differentially. By topographically mapping the hits and misses across the 16 channels it should be possible to determine whether these responses have different cortical distributions, and if they do, how this relates to the discrimination process.

Principal component analyses of the NERP data from each menthol comparison have been conducted employing both the correlation and the covariance matrices. These analyses are currently under review to determine their utility in the quantification of the cognitive component of the NERP.

C. Plans: Continue analyses of the electrophysiological (NERP) and psychophysical data from each of the three menthol discriminations. Following the testing of the new gas supply cabinet of the olfactometer, plans are to: 1) retest some subjects in the NM/SM comparison switching the target and the standard stimuli, and 2) increase the level of difficulty of the natural/synthetic mixture vs synthetic menthol (MM/SM) comparison in order to determine the limits of the cognitive NERP technique for discriminating differences among flavorants. Further plans include the testing of (+) - isomenthol, d-menthol and other yet-to-be determined menthol-like compounds with the goal of developing an index of menthol discriminability.

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PROJECT NUMBER: 1702
PROJECT TITLE: Optical Processing and Aerosol Research
PROJECT LEADER: K. A. Cox
PERIOD COVERED: January, 1989

I. PACK INSPECTION

A. **Objective**: Develop and implement a method for the on-line inspection of cigarette packs.

B. **Results**: A digital circuit has been designed to activate a camera to grab an image in response to an external stimulus. The circuit is needed for on-line operation of the PM pack inspection system. The circuit is currently being built.

The feasibility of significantly increasing the execution speed of the PM inspection algorithm through the use of custom designed hardware is being investigated. The microprocessor currently under consideration is the high speed Motorola 68020 CPU. An effort is being made to obtain a software package which would allow simulated execution of the algorithm on a 68020 system. This will permit identification of the bottlenecks in the execution and provide estimates of the amount of parallelism needed to execute the algorithm at speeds sufficient for high speed print inspection at the printer.

II. INDIVIDUAL CIGARETTE INSPECTION

A. **Objective**: Develop methods for the on-line inspection of individual cigarettes.

B. **Results**: The specifications for the acousto-optic cell needed for the on-line cigarette scanner were finalized and the cell ordered. The design of the scanner's optical system is underway.

An invention record concerning an individual finished cigarette inspection system was filed. An attorney from Fish and Neave was briefed on the details of the technology disclosed in the invention and is drafting a patent application.

C. **Plans**: Complete the design of the optical system for the AO scanning system.

III. INSPECTION OF INCOMING MATERIALS

A. **Objective**: Develop and implement systems for the off-line QA inspection of cut closure stamps and packaging blanks.

B. **Results**: Refinement of the method for the inspection of packaging blanks is underway. Software has been developed in order to permit an operator the flexibility of selecting particular regions for inspection, and the alignment algorithm has been improved to

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permit subpixel alignment. Additionally, the capability for the acquisition of colored images has been added in order to provide optimum contrast between the lettering and the background.

C. **Plans**: Complete the refinement of the packaging blank inspection software. Install the software on hardware selected by IS.

IV. AEROSOL RESEARCH

A. **Objective**: Develop and characterize a laboratory aerosol generator capable of producing highly concentrated aerosols.

B. **Results**: The design of a laboratory aerosol generator with the capability of producing aerosols with concentrations and mean sizes similar to the tobacco smoke aerosol is nearing completion. The generator will produce aerosols by homogeneous nucleation initiated by extremely rapid cooling of both single and multicomponent vapors.

A new light extinction flow cell has been designed, fabricated and installed in the aerosol measurement system. Following testing with previously characterized tobacco smoke aerosols, the system will be used for the measurement of aerosols produced by the laboratory generator.

C. **Plans**: Test the aerosol measurement system by carrying out measurements on tobacco smoke aerosols.

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PROJECT NUMBER: 1704
PROJECT TITLE: Supercritical Fluid Processes
PROJECT LEADER: T. M. Howell
PERIOD COVERED: January, 1989

I. LOW NICOTINE

A. **Objective**: Develop alternate on-line nicotine monitors for the ART process.

B. **Results**: New O-rings for the CD cell continue to hold up. The prototype CD unit from Hinds International was received and installed on the micro-extraction system. Initial check-out procedures indicate the instrument is performing to expectations. Preparations are being made to transfer the unit to the BHPP as soon as possible.

C. **Plans**: This work is ongoing.

II. LOW NICOTINE

A. **Objective**: Develop second generation processing for ART.

B. **Results**: Possible pathways for scaling the liquid adsorber from the laboratory to the pilot plant are still being explored. PM joined the Separations Research Program at the University of Texas at Austin in order to make use of their extensive research in this area. A visit to Austin by PM scientists produced valuable information about column design and about the research capabilities of this outside resource.

C. **Plans**: This work is ongoing.

III. LOW NICOTINE

A. **Objective**: Support to ART commercial plant.

B. **Results**: In order to properly address the corrosion aspects of the ART process, CO_2-water solubility data was calculated over the range of 10°C to 60°C and from 200 psi to 4000 psi. This data was used to form a recommendation on let-down procedures which could be incorporated to prevent the formation of free water within the extraction loop. Eliminating the presence of free water will minimize chances of structural failure due to chloride induced stress corrosion cracking.

C. **Plans**: This work is ongoing.

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PROJECT NUMBER: 1708
PROJECT TITLE: Physical Chemistry and Process Monitoring
PROJECT LEADER: J. L. Banyasz
PERIOD COVERED: January, 1989

I. **OPERATIONS SUPPORT** (J. Crump and A. Closter, in collaboration with the Applied Technology Group)

A. **Objective**: Determine the effect of particle size distribution on dynamic viscosity changes that occur in PVA tipping adhesives.

B. **Results**: The roller speed test at Stockton Street, as reported last month, was continued. The results to date are in accordance with the predictions based on simulator data from the laboratory which indicate that reducing roller idling speed reduces the rate of viscosity increase in the glue pot.

C. **Plans**: The roller speed study at Stockton Street will be completed.

II. **OPERATIONS SUPPORT** (P. Henderson, in collaboration with the Applied Technology Group)

A. **Objective**: Characterization of inks.

B. **Results**: The operating parameter of current tax stamp inks with regard to color, viscosity, and set-off have been established and specifications established. Vendors have been requested to furnish water or alcohol based inks meeting these specifications. The work on establishing the operating window of currently used monogram inks continues.

C. **Plans**: The characterization of monogram inks will continue. New tax stamp inks will be tested as they become available.

III. **LOW DENSITY RODS** (S. Ganeriwala)

A. **Objective**: Compare compression properties of low density and control rods.

B. **Results**: Compression measurements on low density and control rods as a function of RH have been initiated.

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PROJECT 1720: 1720
PROJECT TITLE: Analytical Microscopy
PROJECT LEADER: V. L. Baliga
PERIOD COVERED: January, 1989

I. LOW SIDESTREAM CIGARETTE PAPERS (SANDERS)

A. Objective: Examine the ultrastructure of selected cigarette papers and paper additives in support of the low sidestream project.

B. Results:

Papers: Papers with 30% dolomite and ~10% K Succinate, and papers with 40% dolomite and ~10% K Succinate were examined by scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS)[1]. There were no apparent differences between the fibers on the outer vs the inner sides of the papers, or between the paper with 30% dolomite and the paper with 40% dolomite. There was no difference in the distribution, size, or shape of the dolomite crystals on the outer sides of both papers as opposed to the inner sides of the papers. The crystals partially covered most of the flax fibers and formed aggregates between the fibrils. Both equant and cubiodal crystals were present and were 0.04μm to 2.0μm in size. The primary elements on both surfaces of the paper were Mg, Ca, and K.

Additives: Chemicals from Ecusta were examined using SEM and EDS. They included 1. $CaCO_3$ slurry, 2. $Mg(OH)_2$ dry and 3. $Mg(OH)_2$ slurry[2]. Crystals from the calcium carbonate slurry were elongated and barrel shaped, 0.35μm by 1.5μm in size. The magnesium hydroxide dry powder formed spherically shaped aggregates from 2μm to 140μm in diameter. Two crystalline types present in the aggregates were an equant shaped particle, 0.1μm in diameter, and a thin flattened particle that was difficult to determine a size range. Since the magnesium hydroxide slurry formed a smooth coating on the sample holder, no crystalline structure could was seen.

C. References:

1. Sanders, K., "Characterization of Papers 8726-36-1 and 8726-36-2," memo to S. Baldwin, December 19, 1988.
2. Sanders, K., "Characterization of Calcium Carbonate and Magnesium Hydroxide," memo to R. Comes, December 21, 1988.

II. SUPPORT TO THE ART PILOT PLANT (SANDERS)

A. Objective: Provide support for the continuing research and development efforts of the ART Pilot Plant.

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B. **Results**:

Corrosion Study: Washers that had been attached to the inside of the absorber lid during two runs were removed and examined to see if tobacco bits were collecting on the absorber surfaces. The control washer had many small cracks in the surface as well as Si-containing contaminants. The absorber lid washer had a coating on its surface. This coating was cracked. The coating contained Al, Mg, Si, P, S, Cl, and K. Scrapings from the absorber also were examined. The scrapings appeared dark brown and sticky. No identifiable tobacco bits were found in either the raw scrapings or scrapings after it had been washed with acetone, ethanol, and water to remove the organic materials. The same elements were found in the scrapings as were found on the absorber lid washers. The scraping samples corresponded to samples examined previously[2]. The largest particle noted within the samples was 50µm in length. This and other particles examined did not exhibit any identifiable tobacco morphology.

C. **References**:

1. Sanders, K., "Analysis of Washers and Scrapings from the ART Absorber Lid," memo to B. Fischer, January 20, 1989.
2. Sanders, K., "Analysis of Extractor and Absorber Wall Scrapings," Memo to B. Fischer, November 29, 1988.

III. RESPONSE TO ANALYTICAL REQUESTS (SANDERS)

A. **Objective**: Provide analytical support to R&D.

B. **Results**: Two samples of encapsulated nicotine bitartrate dihydrate, were received to determine the structure of the crystal and the extent of the encapsulation. The samples consisted of large sticky aggregates of crystals. The aggregates of sample 8768-5-2 were more difficult to separate than sample 8768-5-1. Both samples contained thin, wafer-like crystalline structures. They were 1µm to 35µm in size from sample 8768-5-1 and 1µm to 23µm in size from sample 8768-5-2. The crystals from both samples resembled those previously described[1,2]. Since the crystalline structure was clearly visible, instead of a smooth surface coating that a capsule would provide, it was suggested that the samples were not completely encapsulated. The encapsulating material appeared to form a glue holding the aggregates of crystals together as demonstrated by stringy lines between crystals in sample 8768-5-2. This was consistent with the observation that the crystals were harder to separate in this sample[3].

C. **References**:

1. Sanders, K., "Analytical Microscopy Request Form," to S. Haut, August 29, 1988.
2. Sanders, K., "Analytical Microscopy Request Form," to S. Haut, September 12, 1988.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

3. Sanders, K., "Analytical Microscopy Request Form," to S. Haut, January 5, 1989.

IV. SAFETY (SANDERS)

A. **Objective**: Serve as the R&D First Aid Team Captain and member of the Emergency Response Team.

B. **Results**:

One ERT first aid class was given on triage and disaster response.

The Emergency Medical Technician course held at R&D was begun.

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PROJECT NUMBER: 1730
PROJECT TITLE: Plant, Cell & Tissue Culture Research
PROJECT LEADER: I. L. Uydess
WRITTEN BY: M. Shulleeta
PERIOD COVERED: January, 1989

I. TOBACCO-IDENTICAL PRESERVATIVES:

A. **Objective**: To develop procedures and to establish microbiological screens for the evaluation of new, nature-identical preservatives as replacements for and/or as adjuncts to propylparaben.

B. **Status**:

1. Phase I Screens

Several shake-flask experiments were conducted to compare the antimicrobial effect of propylparaben (PP) in two different carriers; triethylene glycol (TEG) and propylene glycol (PG). Stock solutions of propylparaben were prepared in 10% TEG and PG and employed at doses of 75 and 150 µg/ml against the target organism, BC-13 (B. coagulans). The results of each experiment indicated that there were no significant differences between the activity of propylparaben in TEG vs PG at the lowest concentration tested (75 µg/ml) and only a marginal increase in effectiveness (~10-15%) using TEG as the carrier for propylparaben at 150 µg/ml. In each case, however, (150 µg/ml PP-TEG vs PP-PG) only about a half-maximal inhibition of growth was obtained.

A number of additional C-9 to C-12 compounds (lacking nitrogen) have been identified by R. Southwick and R. Izac as logical candidates for screening in the alternate preservatives program. The selection was based upon the observations made thus far regarding the efficacy of the C-9 thru C-12 fatty acids. The compounds selected include:

a. norsolanadione
b. 3-phenylproprionic acid
c. allyl caproate
d. carvacrol
e. methyl-2-nonenoate
f. ethyl phenylacetate
g. solanesol
h. decanol
i. β-cyclocitrylidene acetic acid (and the sodium salt)

2. Phase III Screens

Two dose-response experiments were performed in 5-liter fermentors in order to determine the minimum concentration of decanoic acid necessary to inhibit the gross chemical changes associated with the laboratory-induced spoilage of SEL. A change in pH was used as a general indicator of the chemical modification of the

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SEL. Individual samples of SEL were also submitted to the Analytical Research Division for specific determinations of nitrate, nitrite, total reducing sugars and selected organic acids. Doses of 0, 150, 300 and 400 µg/ml decanoic acid (in PG) were evaluated. The results demonstrated that 150 µg/ml decanoic acid inhibited major chemical changes for up to 12 hours. However, 300 µg/ml of decanoic acid was required to inhibit changes in pH for 24 to 48 hours, or longer (the analytical data were not available at the time of this writing).

C. **Conclusions**:

1. Relatively low doses of decanoic acid (~150 µg/ml) can be employed to inhibit the progression of major chemical changes in SEL for 8 - 12 hours, but higher doses (in the order of 300 µg/ml) are required to inhibit the progression of spoilage for 24 hours or longer based upon a change in pH.

D. **Plans**: **February, 1989**

1. Initiate Phase I screening of new preservative candidates.

2. Continue to optimize the Phase III fermentor screen.

3. Conduct additional Phase III screens of decanoic acid, propyl-paraben and sorbate as individual compounds and in mixtures.

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PROJECT NUMBER: 1752
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke
PROJECT LEADER: G. Vilcins
PERIOD COVERED: January, 1989

I. THERMOGRAVIMETRIC ANALYSIS (TGA)

A. **Objective**: To analyze carbon and low side stream smoke cigarette paper by TGA.

B. **Results**:

Four new carbon samples with high iron content, Vantage Excel Cigarette paper and related papers were analyzed using TGA.

Two of the new carbon samples containing 60 percent and 70 percent iron content actually gained weight due to the oxidation of the iron. The predominant reaction in the samples containing 20 percent and 30 percent iron was combustion.

Low side stream smoke cigarette papers with known compositions were used to develop a method for the determination of magnesium hydroxide and calcium carbonate content in the Vantage Excel cigarette paper. Based on this method the concentrations of magnesium hydroxide and calcium carbonate in the Vantage Exel cigarette paper were 24 and 17 percent, respectively.

C. **Conclusions**: By decomposing all or nearly all the cellulose in low side stream smoke cigarette paper isothermally at a temperature which will not decompose magnesium hydroxide, quantitative analysis can be achieved for it in any low side stream smoke cigarette paper.

D. **Plans**: The work is still in progress.

E. **References**:

Chung, C., PM Notebook 8752, pp. 26-48.

II. PYROLYSIS-GC/MS OF MENTHOL RELEASE POLYMER

A. **Objective**: To examine the release of menthol at a range of temperatures under Curie-Point Pyrolysis conditions.

B. **Results**: Polymic was pyrolyzed at 9 temperatures ranging from 160°C to 920°C. The identities of the volatile gases produced were determined by GC/MS, and estimates of the amount of menthol and menthenes released were determined.

C. **Conclusions**: Conclusions are still being drawn, although preliminary data indicate that menthol release increases with increasing temperature up to ~300°C, where the release levels off until it begins to drop slowly at ~650°C. Menthenes begin to

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appear in significant quantities at ~250°C, and then gradually level off between ~450° and ~650°C. The release of menthenes maximizes at ~750°C, and then drops again. The ratio of menthol to menthenes peaks at the 268°C pyrolysis temperature, although the amount of menthol released at that temperature is only ~60% of the maximum plateau amount of menthol released (at pyrolysis temperatures of~350°C to 600°C).

D. **Plans**: A memo will be written reporting details of the study.

E. **References**:

Magin, D., PM Notebook 8534, page 200.

III. SMOKE ANALYSIS

A. **Objective**: The effect of DIET expansion on the smoke chemistry of ART filler.

B. **Results**: The 100% ART-DIET cigarette, compared with the 100% ART FEED cigarette, delivered to mainstream smoke more pyrazines, phenolics, sugar-related degradation products, isovaleric, and 2-methylvaleric acids; less volatile pyridines, beta-methylvaleric, and 2-methylbutyric acids. The expanded filler delivered comparable amounts of higher fatty acids, simple aromatics, aromatic acids, and tobacco essential oil-related products. The ART-DIET filler shared some characteristics of ET: high in ammonia and low in total reducing sugar. The occurrence of browning reactions prior to and during smoking is more evident than in the previous study.

C. **References**:

Hsu, F. and A. Oti, "Smoke Chemistry - ART/DIET," memo to B. Handy, January 20, 1989.

IV. MASS SPECTROMETRY OF LIPID MATERIALS

A. **Objective**: To develop methodology, utilizing soft ionization and tandem mass spectrometry, for characterization of complex lipid mixtures.

B. **Results**: Work with lipid extracts from tobacco before and after ART processing, samples of residue from the ART process, and known lipid mixtures continues. Evaluation of data obtained from analyses performed using the MS-50 triple analyzer mass spectrometer at the Midwest Center for Mass Spectrometry has revealed much useful information. However, considering the exploratory and developmental nature of the experiments more analyses will be required to confirm and explore potentially significant observations.

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C. **Plans**: Experiments are being designed to explore potentially significant data.

D. **References**:

Jensen, N., PM Notebook 8620, pp.44 -64.

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PROJECT NUMBER: 1757
PROJECT TITLE: Analytical Flavor Specifications
PROJECT LEADER: M. L. Zimmermann
PERIOD COVERED: January, 1989

ANALYTICAL FLAVOR SPECIFICATIONS

A. **Objective**: To develop analytical and sensory specifications for incoming flavors and materials for use at the Flavor Center and other QA facilities. To provide analytical certification on export flavors manufactured at the Flavor Center for FRG compliance.

B. **Results**:

Seven individual PMI samples with unique Direct Material Codes have been released with final certificates of compliance to FRG requirements. Under current guidelines, an additional eight production samples for these same DM codes were also released. A total of six unique PMI samples are currently 'on-hold' and awaiting certification. The source for the contaminant found in three of these materials has been isolated to multiple items. The vendors involved have been contacted and one has confirmed our findings. Communications with their technical representatives are continuing to address this problem.

The halt in export flavor production during the December shutdown allowed a short return to the flavor specifications program. Two-thirds of the materials for the second vendor were examined for final specification purposes. Numerous discrepancies were found in both the nature and the purity of the materials. These problems will be brought to the attention of the vendor.

Assistance has been provided to develop an HPLC procedure for optical brighteners.

C. **Plans**: Continue the certification of the PMI samples and finish the specifications for the second vendor.

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PROJECT NUMBER: 1758
PROJECT TITLE: Tobacco Cell Wall Research
PROJECT LEADER: G. H. Bokelman
PERIOD COVERED: January, 1989

I. SIDESTREAM REDUCTION (S. Baldwin, S. Tafur, B. Rogers and G. Bokelman)

A. Objectives: (1) Prepare and evaluate, for reduction in sidestream smoke, paper handsheets that contain coatings and/or inorganic fillers with different physical and chemical properties and (2) prepare mono- and bilayer cigarette papers on the pilot-scale paper machine at the University of Maine.

B. Results: Sixteen preliminary disclosures for consideration of patentability were prepared concerning sidestream reduction (1).

Samples of a bilayer paper (30% $CaCO_3$/30% $MgCO_3$, 35.3 g/m^2 basis weight) produced in December at the U. of Maine were sized with 12-13% potassium succinate and cigarettes were made both with the $MgCO_3$ layer on the inside and the outside of the cigarette rod. These samples were submitted for sidestream light extinction analysis to check the paper's performance in both configurations.

A variety of bilayer handsheets all produced cigarettes with at least a 50% reduction in sidestream smoke, relative to handmade Marlboro controls. The bilayer papers were 50:50 combinations of $MgCO_3/CaCO_3$, $Mg(OH)_2/CaCO_3$ and $MgCO_3/Mg(OH)_2$. Flow characterization studies of bilayer $MgCO_3/CaCO_3$ handsheets and machine-made paper showed the flow to be linear with pressure drop, indicating true porous flow. Flow profiles were also the same from both sides of the papers.

A special set of handsheets prepared by B. Rogers was received which provided a comparison of paper configurations similar to that of a double-wrapped TRIM V prototype. Single handsheets comparable to both papers of a double-wrapped model, bilayer handsheets, and composite monolayer handsheets were used to prepare three handmade cigarette samples. Sidestream light extinction data showed the bilayer configuration gave the best sidestream visibility reduction. Based on the preceding information, it was requested that a number of mono- and bilayer cigarette papers be prepared by David Kraske and his group at the U. of Maine (2).

A very significant improvement in paper run-ability was achieved at the U. of Maine when a vacuum pick-up bar was installed behind the felt at the couch. As a result, paper breaks originating at the couch decreased dramatically. In the near future we hope to be able to produce cigarette papers with basis weights as low as 18g/m^2.

Thermal gravimetric analyses were obtained for four inorganic fillers: $MgCO_3$, $CaCO_3$, $Mg(OH)_2$, and a precipitated derivative of dolomite. The transition for $CaCO_3$ to CaO occurs at about 770° C while that of $Mg(OH)_2$ to MgO occurs at about 410° C, which is in the same region as the thermal decomposition of cellulose. $MgCO_3$ also undergoes a transition (to MgO) in the range of 400° - 500°C

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The transitions for $MgCO_3$ and $Mg(OH)_2$ may tend to retard the decomposition of cellulose, thereby contributing to a reduction in the burn rate. Both the $CaCO_3$ and $Mg(OH)_2$ transitions were lowered by 30° in the coprecipitated derivative of dolomite, which might be advantageous for sidestream reduction.

A moderate reduction in sidestream visibility was achieved with cigarette papers coated with ART tobacco solubles. However, based on the amount of tobacco solubles added to the paper, the calculated quantity of potassium on the paper was not considered high enough to obtain good fluxing. Therefore, additional papers were prepared with potassium citrate added to the tobacco solubles solution used to coat the papers. Handmade cigarettes were prepared and submitted for sidestream light extinction measurements.

A non-analysis agreement with Union Carbide was signed by PM in order to obtain a sample of their proprietary molecular sieve "Smellrite". This product is touted as a highly effective odor adsorbent which is not deactivated by water and is thermally stable up to 800° C. A half pound sample of Smellrite has been received and requests have been submitted for incorporating this material in cigarette paper handsheets. Also, a mixture of adipic, glutaric and succinic acid has been obtained from Du Pont for evaluation as a potential fluxing agent.

The paper database was completely updated and data were both added and edited. A technician (W. Suiter) was trained on the use and maintenance of this database.

C. **Plans**: A detailed set of plans was prepared for sidestream reduction research during the first half of 1989 (3). Some of the major topics in these plans include: (1) further development of the bilayer concept for effectiveness in sidestream reduction, (2) execution of a statistically-designed experiment to quantify the relative effects on sidestream reduction of porosity, particle size/surface area of filler, and content of fluxing agent, and (3) comparison of machine-made cigarettes prepared using double wrapping, bilayer papers and laminated papers for subjectives and effectiveness in sidestream reduction. With regard to short term plans, hand-made cigarettes will continue to be prepared from numerous types of handsheets for evaluation of sidestream reduction and a memo will be prepared documenting the use of our in-house paper database.

D. **References**:

1. Palmer, Art, memo to Frank Friedman, "New Disclosures Acknowledgment (D479-D494)," January 5, 1989.

2. Bokelman, G. H., memo to R. N. Ferguson, "Results from January Trip to the University of Maine," January 23, 1989.

3. Bokelman, G. H., memo to R. N. Ferguson, "Project 1758 Plans on the Sidestream Reduction Program for the First Half of 1989," January 5, 1989.

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Source: https://www.industrydocuments.ucsf.edu/docs/qtxm0000

PROJECT NUMBER: 1902
PROJECT TITLE: Tobacco Microbiology
PROJECT LEADER: D. J. Ayers
WRITTEN BY: D. K. Chadick
PERIOD COVERED: January, 1989

I. HOGSHEAD, BOX, AND BALE STUDY

A. **Objective**: Microbially analyze burley tobacco stored in hogsheads, boxes, and bales as part of a three year warehouse storage study and to determine the differences, if any, in the microbial numbers from the different containers.

B. **Results**: Grade B57 from the 1986 crop has been examined. The bacterial count from box 6 was above the laboratory standard for burley tobacco; however, the remainder of the microbial numbers from other samples were within the laboratory limits (1).

C. **Plans**: This is a continuing study.

D. **Reference**:

1. Crockett, E. A. Notebook #8681, pp. 176-178.

II. ALTERNATE HUMECTANT PROGRAM

A. **Objective**: To determine the differences after warehouse storage, if any, in the microbial numbers between RL-TC, RL-150B, and RCB sheet material made with PG/G plus propylparaben (control) and Isosweet plus potassium propylparaben (test).

B. **Results**: There were no differences in the microbial counts from the test vs. control sheets after nine (RL) and eight months of storage (RCB) (1).

C. **Plans**: A memo is in preparation.

E. **Reference**:

1. Jones, J. Notebook No. 8590, p. 87.

III. ESTABLISHMENT OF A STANDARD OPERATING PROCEDURE (SOP) FOR HIC-2 (HUMIDAT)

A. **Objective**: To develop a procedure for the rapid determination of OV from various tobaccos and tobacco products.

B. **Results**: Several experiments designed to validate the use of the Humidat in determining OV were completed this month. Standard curves have been made for bright, burley, and oriental tobaccos (1,2).

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C. **Plans**: Studies are continuing with recon (RL-TC,150B, and RCB) and a SOP will be issued.

D. **References**:

1. Gaines, O. Proposed Procedure for the Evaluating the HIC-2 (Humidat). Memo to D. Ayers; 1988 December 8.

2. Gaines, O. PM Notebook No. 8690, pp. 95-97.

IV. SALMONELLA/MICROSOME (S/M) ASSAY: DECANOIC ACID STUDY

A. **Objective**: To determine if the IT CSC specific activity (S.A.) of 1R4F cigarettes is altered when decanoic acid is oversprayed onto the filler.

B. **Results**: Decanoic acid was dissolved in ethanol at concentrations of 25, 50, and 100 mg/ml and oversprayed onto 1R4F filler. The IT CSC S.A.s from the cigarette samples were compared to those of the solvent and 1R4F controls. The results showed that there were no statistically significant differences among any of the test samples and the controls (1).

C. **Plans**: There are no plans for further testing in this area.

D. **Reference**:

1. Thompson, L. H. Notebook No. 8731, pp. 42-51.

V. S/M ASSAY: YEAST ENCAPSULATED CIGARETTES (YEPO)

A. **Objective**: To evaluate IT CSC S.A. from one control and two test cigarettes which were prepared by incorporating into the filler (full-flavored blend) yeast encapsulated material; i.e., peppermint oil.

B. **Results**: The YEPO cigarettes were prepared at two concentrations, 110.2 and 1102 ppm for S/M evaluation. The results from four replicate smokings of each sample indicated that there were no differences in S.A. among the test samples or the control (1).

C. **Plans**: There are no immediate plans for further tests in this area.

D. **Reference**:

1. Thompson, L. H. PM Notebook No 8731, pp. 52-59.

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PROJECT NUMBER: 1904
PROJECT TITLE: Tobacco Physiology and Biochemistry
PROJECT LEADER: H. Y. Nakatani
PERIOD COVERED: January, 1989

I. LOW NICOTINE STUDY

A. **Objective**: To investigate the biochemistry of the nicotine biosynthetic pathway at putrescine N-methyltransferase (PMT) and specifically to isolate PMT from tobacco root extracts.

B. **Status**: Root extracts (PM 92-99) from the remainder of the group 15 hydroponically grown burley 21 tobacco plants were processed to the 40-65 % ammonium sulfate stage and stored at -80°C.

Aliquots from one of the phenyl-Sepharose columns (M-4) were put on a S-adenosylhomocysteine (SAH) affinity matrix (blocked with mercaptoethanol) for the continuation of the purification of PMT. PMT is eluted from this column with 1 mM SAM. Some PMT and other proteins, which are bound tightly and require 0.5 M NaCl for their elution, are left on the column (5). After equilibrating the column with dialysis buffer, an additional 100 ml aliquot of a partially purified PMT (M-4 phenyl-Sepharose) sample was applied to the SAH column and the PMT is again eluted with 1 mM SAM (3,5). PMT active fractions from the SAH column are pooled and stored at -80°C.

Three SAM-eluted PMT samples from the SAH columns were further purified by application to a ω-aminoethyl Agarose (AEA) matrix. The peak fractions having PMT-activity upon elution with 5 mM putrescine were pooled and frozen for further studies (4). The PMT after the three column purification scheme (phenyl-Sepharose, SAH-affinity and AEA-affinity columns) with these samples had a specific activity of 4,500 - 10,000 units (nmoles/mg protein at 30°C for 30 min.) (2,4,5). The pooled samples after the AEA-affinity column showed an increase in specific activity of 206-460-fold compared with the ammonium sulfate stage. Our initial objective to obtain 100 µg of protein with an increase in specific activity of 1000 over the ammonium sulfate stage was met with AEA2 (>300 µg protein, >2000-fold increase in specific activity). These samples were stored for further examination. Samples from the SAH purified stage (SAH1 and SAH4) were submitted to Dr. Bruce Davies for ^{3}H-SAM photoaffinity labeling studies.

A Rotofor experiment was conducted with an SAH1 (M1) sample (3). The SDS-PAGE profile showed for the peak fractions polypeptides in the 50-65 kD molecular weight region with less intense bands in the 40 kD region (3).

Dr. Walter Hempfling applied PMT-active samples from the SAH column (not peak samples) to a Bio-Gel P200 gel filtration column. The SDS-PAGE profile of the fractions eluting around the peak showed polypeptides in the 60 kD region and more intense bands in the 40 kD region (3).

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The Protein-Gold (PG) reagent procedure (developed by Integrated Separations System [ISS]) appears to offer the possibility to assay small amounts of protein (4). The standard curves for this procedure (using γ-globulin as a standard) were reproducible and linear over a range of 0-20 µg/ml. Samples to be assayed were dialyzed (tubing or Prodialyzer, ISS) or desalted first (PD-10), since salt, SDS, and reducing agents interfere with the assay. Dialysis tubing or PD-10 columns (against deionized water) worked best to eliminate interferences. The Prodialyzer was ineffective in removing interfering components even after 6 hrs.

C. **Plans**: Process roots from group 16 to the 40-65% ammonium sulfate stage. Prepare enriched PMT samples from the ammonium sulfate stage by using the three columns: phenyl-Sepharose, SAH, and AEA. Continue the evaluation of the Protein-Gold reagent assay method for determining low concentrations of protein. A method to precipitate the protein (concentration) with sodium deoxycholate is being examined which may eliminate the necessity to dialyze or de-salt the protein (PD-10) sample.

Evaluate other classical chromatographic methods (in collaboration with Dr. W. F. Hempfling) to yield a homogeneous PMT preparation. Continue Rotofor experiments and photoaffinity labeling studies (in collaboration with Dr. B. Davies).

D. **References**:

1. Dunn, R. Notebook No. 7899.
2. Malik, V. Notebook No. 8542.
3. Davies, S. Notebook No. 8694.
4. Yu, T. Notebook No. 8381.
5. Mooz, E. Notebook No. 8599.

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PROJECT NUMBER: 2106
PROJECT TITLE: Cigarette Performance and Design
PROJECT LEADER: R. W. Dwyer
PERIOD COVERED: January, 1989

CIGARETTE DESIGN MODELING (B. Dwyer)

A. **Objective**: Develop computer-based cigarette-design models for modifying the properties of current products and for designing novel ones.

B. **Results**: A Fortran-based model has been developed which allows one to design cigarettes to meet tar, puff count, and cigarette RTD targets. The model allows the user to explore the effects of filter construction, ventilation, tobacco-rod construction, and burn rate on tar delivery and puff count. At this point, the computer model includes routines for conventional and concentric CA filters.

C. **Plans**: We are developing routines for handling paper, CA-web, and polypropylene filters. These should be incorporated into the model within two months.

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PROJECT NUMBER: 2500
PROJECT TITLE: Fundamental Chemistry
PROJECT LEADER: J. I. Seeman
PERIOD COVERED: January 1989

I. FLAVOR/ODOR CHEMISTRY (Houminer, Paine)

A. Objective: To develop new technologies for smoke deliveries of desired flavorants, especially menthol; to prepare new substances for flavor/odor evaluation, especially as they relate to SS and ashtray odor.

B. Results and Plans: Poly(menthyl-1,1-dimethylallyl carbonate) (polyMDAC) is being investigated as an alternative source for menthol release. Experiments are being performed to establish the backbone structure of polyMDAC and alternative polymerization initiations are being examined.

The preparation of three polyalkylpyrazines has been completed: 2-propyl-6-methyl-, 2-isopropyl-6-methyl- and 2,3-dimethyl-5-isopropylpyrazine. While these were made for laser jet studies, the latter two were found to have a pronounced 'ashtray' odor.

II. REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS (Howe, Secor, Seeman)

A. Objective: To develop techniques to remove nicotine and other tobacco alkaloids from aqueous tobacco processing fluids to the exclusion of all other components.

B. Results and Plans: A series of fourteen nicotine extractions was run using the Sepracor 2-module system. Nicotine analyses were performed by C. Tatum. The nicotine was transferred to a single reservoir. The net concentration effect was 18.8 times the starting concentration. The overall efficiency of extraction was 91%. Approximately 20% of the nicotine was held-up in the membrane, while no nicotine accumulated in the organic solution. BIS was the LIX.

Additional effort to prepare proprietary LIXs is in progress. 186 g of the 2-ethylhexyl monoester of dipropionyl tartaric acid was prepared and 118 g were sent to Sepracor. The related 2-octyl ester was prepared and shows excellent phase properties.

III. INORGANICS AS NOVEL TOBACCO MATERIALS ADDITIVES (Fournier, Paine)

A. Objective: To develop inorganic materials for novel applications for reduced sidestream and enhanced subjectives in cigarettes and for required properties in novel smoking materials.

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B. Results and Plans: Persuant to a request from A. Kallianos, 1 mole of calcium ethoxide in anhydrous ethanol was prepared. Various magnesium phosphates, designed to occur in, or be transformed to, a suitable particle size, were prepared. Plans are being developed for the use of inorganic synthesis to elucidate the influence of surface area and morphology on SS properties.

IV. MISCELLANEOUS (Secor, Seeman)

A. Results and Plans: 1.0 g samples of dipentyl α-, β-, and γ-cyclodextrins have been prepared and 0.5 g of each sent to D. Armstrong (University of Missouri) to be tested as gc coatings. Acetylation of these materials is now in progress. Initial gc results indicate that enantiomeric separations are feasible.

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PROJECT NUMBER: 2501
PROJECT TITLE: Smoke Chemistry
PROJECT LEADER: R. Comes
PERIOD COVERED: January, 1989

I. SIDESTREAM SMOKE

A. **Objective**: Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream gas phase and semivolatiles; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of proprietary products.

B. **Status**: 1.) a) Sidestream gas phase and semivolatile analyses continue. A new Tekmar system has been installed and is being evaluated for gas phase studies. b) A sniffing port has been installed on a Varian Vista 6500 gas chromatograph. c) Work has begun on the development of methods for quantitation of CO, CO_2, water and neophytadiene as indicators of total sidestream and mainstream smoke. These methods should permit the determination of "differences" between cigarettes made with conventional wrappers and those with wrappers yielding reduced sidestream visibility. d) Work has continued to bring the new total recovery smoking machine on-line. The system continues to have flow control and leak problems. Initial plans have been formulated to investigate C-14 labelled reference and reduced visibility cigarettes using this smoking machine.

2.) a) Construction of a new 1-port sidestream visibility monitor is nearing completion. This system is modeled after the long-used system in Development. b) The 8-port sidestream visibility apparatus is fully operational. Several control and reduced visibility models have been run. Data appears to compare favorably with that generated on the 1-port system although, as expected, the absolute numbers are different.

3.) A new study has begun to attempt to evaluate cigarette wrappers utilizing pyrolysis/gc/ms. Selected samples of reference and low sidestream papers have been pyrolyzed at 350°C.

C. **Plans**: 1.) a) The old Tekmar unit will be returned to the factory for upgrading, repair and refurbishing. It will then be returned to us and used in additional sidestream studies. Experimental and reference models will be evaluated for sidestream gas phase and semivolatiles as requested. b) The sniffing port will be used for investigations of sidestream smoke components of importance in the odor and irritation area and for evaluation of flavor materials in the development of a non-burning (swept) smoking device. c) Selected cigarette models will be studied to see if ratios of gas phase to particulate and total sidestream smoke to total mainstream smoke change as wrappers that are useful in sidestream reduction replace conventional wrappers. d) Efforts will continue on construction of the total recovery smoking machine. Checkout and data comparison with that generated from previous machines

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will be required before use in total distribution studies in the sidestream area are possible.

2.) a) Data from the new, 1-port visibility apparatus will be compared with that from the Development system. When acceptable, the new apparatus will be placed into routine operation to alleviate some of the current sample backlog and to aid in future sidestream efforts. Development plans are underway to construct a third system. b) Evaluation of the 8-port apparatus will continue to develop a sample history and to determine system reproducibility. An operation and procedure manual is being written in anticipation of turning the system over to CI with its ultimate use as a production monitor of visibility in the Louisville factory. Plans for a totally new 8-port system are currently being formulated.

3.) Development of a method and conditions for pyrolysis/gc/ms paper analysis will continue. Differences in compounds produced from conventional cigarette papers vs papers for reducing visible sidestream smoke will be identified. Quantitation attempts will follow.

II. SIDESTREAM SMOKE CHAMBER

A. **Objective**: Design and construct an environmentally controlled chamber to measure selected components of sidestream smoke.

B. **Status**: a) The modifications to the outer office areas have been completed. One office has been occupied by personnel who will be directly responsible for day-to-day chamber operation. The other office will be utilized in subjective studies directly and indirectly related to chamber operation. b) Some of the instrumentation ordered for chamber studies has been received. It is currently being set-up for evaluation.

C. **Plans**: Instrumentation will be used to generate data under normal conditions prior to installation in the chamber upon its completion.

III. MISCELLANEOUS

1. Nicotine analyses have been and continue to be run in support of the Sepracor nicotine removal efforts for the ART project.

2. Routine gc/ms and pyrolysis/gc/ms continues as requested.

3. Neutron radiography (at U.VA.). a) Experimental Philip Morris cigarettes continue to be studied by this technique as requested. b) The programs and techniques for measuring the build-up and movement of condensible materials in a burning cigarette have been reviewed and a successful test run made on a Marlboro 100. The results were similar to those obtained in previous experiments. Runs on 17mm circumference models will be made soon to see if

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differences are evident between cigarettes of varied circumference.

4. a) The schlieren imaging system is being reassembled to reestablish this technique as a routine procedure in the sidestream program. b) Plans are underway and paperwork is in progress to purchase a new infrared camera for use in the sidestream program. c) The TEOM mass monitor has been assembled and tested. It works without any apparent problems. Some computer software routines need to be written.

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PROJECT NUMBER: 2525
PROJECT TITLE: Tobacco Chemistry
PROJECT LEADER: R. R. Izac
PERIOD COVERED: January, 1989

I. NATURAL PRODUCTS CHEMISTRY

A. **Objective**: To isolate, identify and/or analyze natural compounds with major emphasis on tobacco and tobacco products.

B. **Results**: 1.) About a gram of solanesol was isolated from Diet solids for AR (H. Randolph) and I. Uydess. 2.) As part of a collaboration with Project 6906, the separation of Burley S1 (BuS1) fraction using Davisil C18 was completed. A filter funnel was filled with the C18 packing and the BuS1 applied. Elution with water and methanol separated the material into two fractions. Five more such washings were needed to remove remaining material--the latter fractions (F2-F7) were combined. The resulting two fractions were submitted to D. Magin for further analysis.

C. **Plans**: Continue separation of BuS1 using other methods.

D. **References**:
1. Izac, R. Notebook No. 8632.
2. Core, M. Notebook No. 8608.

II. GREENHOUSE STUDIES

A. **Objective**: To maintain the R&D greenhouses, to conduct plant research studies and to provide greenhouse-grown tobacco materials for support of other R&D programs.

B. **Results**: 1.) The preparation of the hydroponic Burley 21 plants been started. These plants are being grown for Project 1904. 2.) The greenhouse maintenance operations including seeding, transplanting, nutrient solution preparation and other cultural tasks were completed. The installation of new vent screens is in progress. 3.) Culture of Dryopteris felix-mas is continuing and several more of these have been ordered. Specific compound of interest (phloroglucinols) to FTR and AR will be isolated from the plants at maturity.

C. **Plans**: Maintain production of fresh root tissue by hydroponic culture.

D. **References**:
1. Bass, R. Notebook No. 8607.
2. West, G. Notebook No. 8559.
3. Izac, R. Notebook No. 8632.

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III. SUPPORT ACTIVITIES

A. **Objective**: To provide requested assistance for special projects.

B. **Results**: 1.) Solanesol was hydrogenated and the resulting octadecylhydrosolanesol purified for AR. The sample is to be used as an internal standard in the analysis of solanesol. 2.) A sample of succinylated nicotine is being chromatographed by hplc for H. Secor. 3.) During the last few months, a total of 469 hand made cigarettes were fabricated for the sidestream control project. 4.) The thirty third Tobacco Workers Conference was attended. As the PM representative, R. Bass attended the Variety Standards Committee meetings. 5.) Filler from the 1988 Burley Tobacco Regional Farm Test were received and processed into cigarettes for flavor panel evaluation.

C. **Plans**: Continue chromatography of succinylated nicotine.

D. **References**:

1. Izac, R. Notebook No. 8632.
2. Bass, R. Notebook No. 8607.
3. Core, M. Notebook No. 8608.

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PROJECT NUMBER: 6502
PROJECT TITLE: Environmental Tobacco Smoke
PROJECT LEADER: C. E. Thomas
WRITTEN BY: M. E. Parrish
PERIOD COVERED: January, 1989

I. ETS STUDIES

A. **Objective**: Develop a method for determining the MS and SS deliveries of solanesol in cigarette smoke for use in the evaluation of solanesol as a marker for environmental tobacco smoke (ETS).

B. **Results**: A SS chamber was made by John Bivins such that the top opening was designed to hold a normal Cambridge filter pad for collecting the SS TPM. The MS and SS TPM pads were extracted using toluene, derivatized using BSTFA and pyridine and analyzed using a 15 M, J&W DB5 capillary column connected to a flame ionization detector. The separation was obtained using a temperature program from 70°C to 300°C at 20°C/min with a final temperature hold of 15 minutes. The internal standard (octadecahydrosolanesol)[1] eluted at 18 minutes and solanesol eluted at 24 minutes. For Monitor 24 the deliveries obtained were: 420 to 510 µg/cigt MS and 130 to 180 µg/cigt SS for a SS/MS ratio of 0.3[2].

C. **Conclusions**: Collection, derivatization, and gas chromatographic conditions have been defined in the procedures for measuring MS and SS solanesol. Sufficient resolution for solanesol and an internal standard have been obtained for MS and SS TPM of Monitor 24 cigarettes.

D. **Plans**: Evaluations to determine the efficiency of collection and recovery studies with known levels of solanesol will be performed.

E. **References**:

1. Izac, R., Personal Communication, January 17, 1989.
2. Randolph, H., PM Notebook #8475, pp. 180-181.

II. MAINSTREAM AND SIDESTREAM SMOKE STUDIES

A. **Objective**: Use the newly developed TDL procedures for the evaluation of MS methanol deliveries of different types of cigarettes.

B. **Results**: The methanol delivery for the control MF blend (#6359) was 240 ± 40 µg/cigt (28 ± 3 µg/puff). The deliveries for the MF blend with standard casing (#6360) and the MF blend with standard casing and A/C (#6361) were 255 ± 18 µg/cigt (29 ± 2 µg/puff) and 260 ± 18 µg/cigt (29 ± 2 µg/puff), respectively.

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C. **Conclusions**: Based on these samples, the MS methanol deliveries were not affected by the casing or A/C flavors applied to the MF blend.

D. **Plans**: MS methanol will be measured for the 100% single blend component cigarettes made in 1988.

E. **References:**

Lipscomb, J., PM Notebook #8703, p. 52.

A. **Objective**: Measure MS ammonia deliveries for ART experimental cigarettes.

B. **Results**: The ART feed control (X6D8DEJ) and the ART extracted filler (X6D8DEK) cigarettes were analyzed for MS total ammonia with the filter plug removed and the ventilation holes taped. The deliveries were 60 ± 15 and 130 ± 20 µg/cigt, respectively. Monitor 24 cigarettes analyzed in similar fashion delivered 56 µg/cigt compared to 30 µg/cigt when smoked with filter intact. These samples also were used to determine the MS total ammonia delivery for the eight puff only. Four cigarettes were smoked with filter intact into the solvent trap to obtain sufficient sample. The eighth puff delivery was 5 µg/puff for the feed control (X6D8DEJ) and 14 µg/puff for the ART extracted filler (X6D8DEK)[1].

C. **Conclusions**: The level of ammonia in the eighth puff of the cigarettes made from ART extracted filler approaches the 17 to 22 µg/puff threshold previously determined using E-55 Bright cigarettes[2]. Serious consideration should be given to reduce the ammonia delivery in the ART cigarettes.

D. **Plans**: The ART cigarettes will be analyzed again with the filters intact so that comparisons can be made with other samples being smoked using normal procedures. Lactic acid has been suggested as a means for lowering the filler pH and reduce the ammonia delivery. These experiments are being conducted using various levels of lactic acid[3].

E. **References**:

1. White, G. and H. Randolph, "Mainstream Ammonia in ART/DIET and ART Feed Filler," memo to B. Handy, January 3, 1989.

2. Parrish, M., Harward, C. and G. Vilcins, "Determination of the Gaseous Ammonia Threshold in Cigarettes Smoke, Part I, Analytical Methodology," Completion Report, June 7, 1985, Acc. No. 85-175.

3. Parrish, M., PM Notebook #8729, pp. 17-19.

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PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
PROJECT LEADER: D. F. Ingraham
PERIOD COVERED: January, 1989

I. PROJECT ART

A. Objective: Provide analytical support to project ART.

B. Results:

A Stabilwax-DB® column from Restek Corporation is currently being evaluated as an alternative to the J&W CAM column, due to long delays in receiving the latter columns. Thus far, the Restek column appears to be satisfactory for the GC determination of nicotine. The optimum linear carrier gas velocity for this analysis was determined to be about 30-32 cm/second, which should be applicable for any 15m x 0.25mm ID, 0.25 µm film thickness column currently in use.

Monitor data collected at BHPP from runs numbered 258 to 279 were subjected to statistical analysis by Dick Jones. The high (unextracted) monitor showed an average value of 2.65% with upper and lower control limits of 2.76 and 2.54, respectively, and an estimated standard deviation of 0.05. The low (extracted) monitor showed an average of 0.068% with upper and lower control limits of 0.078 and 0.057, respectively, and an estimated standard deviation of 0.005. These data suggest that the extracted samples can be reported to three decimal places and the feed samples to two decimal places. The control charts showed a fairly random distribution around the mean for the low monitor, but several data points in a row above and below the mean for the high monitor.

C. Plans: Continue analytical support on an as needed basis. Continue to run and analyze the monitor data at BHPP. Investigate any analytical differences between the 5880A and the 5890A GC's.

II. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIAL

A. Objective: To provide headspace analyses for residual solvents from packaging materials and develop a QA method for the routine analysis of packaging materials.

B. Results:

Approximately 20 samples were analyzed by GC/HS this past month for residual solvent content. Several of these samples were also run by GC/FT-IR/MS for confirmation or identification of unknown solvents.

Phone support and a sample of our standard was provided to one of our vendors (Golden Belt). A standard solution was also provided to QA. Don Simmons (QA) was given a quick introduction to the

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GC/HS analysis and provided with sufficient information to get started when his equipment arrives. Don will be assuming responsibility of analyzing the routine samples in QA.

C. **Plans**: We will continue to analyze non-routine samples and provide support to QA as needed, including further training of the analyst in QA.

III. MATERIALS EVALUATION

A. **Objective**: To identify components of commercial products prior to their use at PM facilities.

B. **Results**: Samples analyzed this month included cleaners, machine parts, lubricants, tube belts, and metal coatings. Recommendations were made for all samples.

C. **Plans**: The analysis of samples for the audit program will begin after receipt of the January report from Purchasing.

IV. RESPONSE TO ANALYTICAL REQUESTS

A. **Objective**: To provide analytical support to R&D and Operations personnel and projects.

B. **Results**:

Analyses and investigations by project personnel during the month of January included:

Several customer complaint samples were analyzed for possible contaminants.

Approximately 100 filter plug samples were analyzed for water, triacetin, and menthol. These plugs, which were submitted by T. Van Auken, contained different levels of triacetin and had been exposed to menthol vapors at different relative humidities. The results showed that both water and triacetin content had an effect on the level of menthol observed, but a simple model could not explain all the data. These data were reported to the submitter for further analysis.

Anethole and ethanol content of Marlboro cigarettes were determined for the Marlboro Standardization Program.

A sample of paraben was received from Park 500 which was determined to have an off odor. Compared to an acceptable control, the sample was found to contain 2,2,4-trimethyl-1,3-dioxolane.

Several tobacco samples were analyzed for calcium by titration. The titration method allowed a faster turnaround and relieved the workload on the AA, where this analysis had been performed in the past.

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PROJECT NUMBER: 6906
PROJECT TITLE: Biological Effects of Smoke
PROJECT LEADER: J. M. Penn
WRITTEN BY: W. R. McCoy
PERIOD COVERED: January, 1989

I. GLUTATHIONE DEPLETION ASSAY (GDA)

A. **Objective**: To determine the time course depletion of GSH by DEM in TA98 cells.

B. **Results**: Percent reduction in the GSH level with DEM treatment versus a DMSO control treatment over time was as follows: 0 time, 3% reduction; 1 hr., 42% reduction; and 2 hr., 55% reduction. The percent reduction in the GSH level versus the control was not due to a decrease in the GSH level with DEM treatment, but was due to an increase in the GSH level with DMSO treatment.

C. **Plans**: Repeat the experiment and include saline control treatments and toxicity evaluations.

D. **Reference**: McCoy, W. R. Notebook No. 8739, pp. 28-30.

II. INHIBITION OF EGF BINDING ASSAY

A. **Objective**: To compare two methods of determining nonspecific binding.

B. **Results**: The standard method is to add one amount of excess cold EGF to each dilution used for determining total binding. The alternate method was to add cold EGF at a fixed ratio of hot to cold. There were only minor differences in nonspecific binding between the two methods. Lundon analysis of this data revealed only one binding site.

C. **Plans**: Begin to develop methods for studying EGF receptor phosphorylation.

D. **Reference**: Patskan, G. Notebook No. 8751, p. 142.

III. BINDING OF PDBu TO 3T3 CELLS

A. **Objective**: To examine PDBu binding with treatment at the time of binding.

B. **Results**: CSC tested at time of binding has shown inconclusive results. There appears to be some inhibition of binding by the CSC solvent, DMSO.

C. **Plans**: To try acetone as a solvent for CSC and to compare 2R1 to X6D5RN.

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D. **Reference**: Vaughan, B. Notebook No. 8747, p. 151.

IV. NICOTINE SPECIFIC MONOCLONAL ANTIBODY

A. **Objective**: To obtain a monoclonal antibody (MCA) against nicotine (NIC-MCA).

B. **Results**: A reaction was carried out to conjugate succinylated hydroxyethylnicotine (SHN) and polylysine (PL) for use as an antigen for screening nicotine-specific MCAs. Spectrophotometric analysis of the reaction indicated that the SHN did not covalently attach to the PL. Analysis of the reaction pH, concentration of precursors, and the reaction temperature indicated that they were as specified in the reaction protocol. Since the reaction is dependent upon the stability of the 1-ethyl-3(3-dimethylaminoproply) carbodiimide (EDAC), a second source of EDAC was obtained and a second reaction performed. The reaction products are currently being analyzed.

C. **Plans**: Complete the synthesis and characterization of PL-SHN reactions. Continue screening hybridoma clones.

D. **Reference**: Davies, B. D. Notebook No. 8638, p. 150.

V. APPROACHES TOWARD PUTRESCINE METHYLTRANSFERASE (PMT) ISOLATION

A. **Objective**: Provide additional experimental approaches to assist in the effort to isolate PMT.

B. **Results**: An experiment was carried out to examine the saturation levels of the photostimulated covalent attachment (PAL) of 3H-SAM to PMT. Specific SAM-PAL labelling appeared to saturate. For the particular PMT preparation used, the maximum labelling saturated at ~0.16 pmol SAM/µg of protein.

C. **Plans**: Repeat and expand additional studies to examine the specificity of PAL labelling. Label a large batch of PMT using an optimal concentration of 3H-SAM and analyze the labelling using both one and two dimensional SDS-PAGE analysis.

D. **Reference**: Davies, B. D. Notebook No. 8638, p. 170.

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PROJECT NUMBER: 6908
PROJECT TITLE: Smoke Condensate Studies
PROJECT LEADER: A. H. Warfield
PERIOD COVERED: January, 1989

I. TSNA PRECURSORS

A. **Objective**: To determine the precursors of MS TSNA.

B. **Results**: Microencapsulated nicotine bitartrate (NBT) has been received from R.T. Dodge Co. Two different versions were prepared which will release nicotine at different temperatures in order to serve as models of unextracted nicotine, suspected as a precursor of MS NNK. The materials are being characterized. X-ray microscopy shows very little difference between the microencapsulated and untreated NBT crystals except for filaments on the former.

Method development needed to measure ^{14}C-TSNA formation in MS smoke from cigarettes containing ^{14}C-nicotine was continued. Organic and aqueous phases from a buffer extract of MS TPM spiked with NNK, NNN and nicotine which were labeled with ^{14}C were analyzed by TLC and/or HPLC with radiochemical detection. Approximately 10% of the labeled nicotine was found in the methylene chloride (MeCl) phase, the remainder being in the aqueous phase. ^{14}C-NNK and ^{14}C-NNN were just detectable in the MeCl phase after concentration. Application of larger samples to the TLC plate resulted in streaking or diffusion.

Oriental (Or) CEL was sprayed onto DBC Bu filler which had been extracted with 5% EtOH/Hexane in an attempt to determine whether pyrosynthesis could be inhibited in a filler which contains minor alkaloids and nitrate but very little nicotine or endogenous TSNA. However, the resulting filler was sticky and clogged the maker when an attempt was made to prepare cigarettes. OrCEL was also sprayed onto an RL made previously from 1/2 the normal level of BuCEL on Bu base web (BW), yielding 1/2 OrCEL on 1/2 BuCEL on BuBW. A significant reduction in MS TSNA was achieved, but the reduction was not as great as when the CELs were premixed before spraying onto the BuBW, suggesting that chemical or physical interactions result when the CELs are mixed. Experimental RLs have also been prepared to test the role of major salt cations of Or tobacco in the reduction of TSNA observed in the mixed RL study. Data on these RLs will be available shortly.

D. **Plans**: The microencapsulated nicotine will be incorporated into filler and MS smoke data obtained in order to determine whether TSNA are formed. Cigarettes prepared from filler containing ^{14}C-nicotine will be smoked and the MS-TPM analyzed for ^{14}C-NNN and ^{14}C-NNK using methods currently under development. Additional studies with OrCEL on extracted fillers are in progress.

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E. **References**:

Haut, S. A. Notebook 8768, p. 15.
Hassam, S. B. Notebook 8712, p. 109.
Hellams, R. D. Notebook 8613, p. 125.

II. ORIENTAL TOBACCO STUDIES

A. **Objective**: To determine if the inhibition of TSNA formation/pyrosynthesis is due to agronomic effects.

B. **Results**: Filler TSNA data for the McNair 373 (bright control) and the three Samsun (oriental) test samples that were grown in Whiteville, N.C., were reexamined, omitting extremely high and extremely low replicates. However, the data still resulted in the conclusion that Samsun tobacco grown under bright conditions and sun-cured produces more endogenous TSNA when not topped than when topped, and much more TSNA when flue-cured than when sun-cured.

A pilot study was initiated to determine whether there is a difference between various types of oriental tobacco in TSNA content and/or MS TSNA delivery. Three types were readily available from another study, and some analytical data were available on these samples: TIU (Turkish Izmir), TSK (Turkish Samsun) and GMU (Greek Macedonium). Filler TSNA data were obtained on these fillers, and alkaloid as well as MS TSNA data are being obtained.

C. **Plans**: Cigarettes have been prepared from fillers obtained in both of the above studies and MS TSNA data are now being obtained. Other data will be obtained from ARD. Interpretation of the results of the U.S. grown oriental study as well as the pilot study of oriental types will be attempted when all the data are complete.

D. **References**:

Keene, C. K. Notebook No. 8754, p. 32.
Haut, S. A. Notebook No. 8768, p. 15.

III. CROSSED SOLUBLES/BASE WEB STUDY (CHEMISTRY)

A. **Objective**: To investigate the smoke chemistry of model cigarettes made from all possible combinations of solubles from bright, burley and oriental tobaccos on base webs from the three tobaccos.

B. **Results**: OrCEL + BuCEL were mixed in equal proportions and sprayed onto BuBW to reproduce the RL previously shown to yield reduced TSNA levels relative to a BuCEL on BuBW control. Appropriate controls were also prepared. The fillers were made into handmade cigarettes and smoked in quadruplicate to collect IT CSCs for submission to Project 6906 for S/M testing. Additional work was done on the electrodialysis equipment to prepare it for service in order to remove specific ions from CEL solutions.

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C. **Plans**: The membrane stack will be installed and the electrodialysis equipment run using a standard salt solution. Plans are being formulated to examine the effects of cations, nitrogenous compounds, sugars, and ammonium salts. Further plans will be considered to design synthetic or reconstituted CEL, remove CEL protein, and use glycosidases and peptidases on CEL and BW. Mixed CEL studies are also being planned.

D. **References**:

Hassam, S. B. Notebook 8712, p. 109.
Hellams, R. D. Notebook 8613, p. 125.
McGee, N. H. Notebook 8743, pp. 7-8.

IV. SUPPORT FUNCTION: CONDENSATE PREPARATION

A. **Objective**: To fabricate cigarettes, perform smokings, and prepare condensate as needed for biological and chemical analysis.

B. **Results**: 2R1 IT CSC condensate was collected and processed for testing in the EGF assay. Machine-made cigarettes containing two different levels of encapsulated yeast, along with a control, were smoked in order to obtain IT CSC to be tested in the S/M assay. Hand-made cigarettes were also prepared from three different fillers submitted by M. Parrish.

C. **References**:

Hellams, R. D. Notebook 8613, pp. 124-125.
McGee, N. Notebook 8743, pp. 6,9.

V. EFFECT OF CASINGS ON TSNA DELIVERY:

A. **Objective**: To determine the effect on MS TSNA of casings or flavors normally included in commercial products but not found in reference cigarettes.

B. **Results**: The following samples were smoked for MS TSNA: 1. full flavored blend with PG/G; 2. full flavored blend PGG/G free with Isosweet; 3. RLTC with PG/G; 4. RLTC PG/G free with Isosweet; 5. RL 150-B with PG/G; and 6. RL 150-B PG/G free with Isosweet. The objective here was to determine whether the sugars and/or cooked flavor used in some of these casings have any effect on TSNA delivery. The data obtained showed no effect of the sugars on NNN or NAT deliveries, but the presence of the added sugars correlated with a marginally significant increase in MS NNK. The reason for the increase was not explained by an examination of analytical data obtained on the fillers. There was no difference in MS TSNA delivery attributable to the 150-B.

C. **References**:

Tickle, M. H. Notebook 8716, pp. 151-152.

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PROJECT NUMBER: 8101
PROJECT TITLE: Cigarette Testing Services Division
SECTION LEADER: Joyce F. Stargardt
PERIOD COVERED: January, 1989

I. MARKET ACTIVITY

A. **Objective**: To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. **Results**:

1. New Brands

Philip Morris will introduce nationally Alpine 80 (Box) and Alpine Lights 80 (Box) cigarettes in February. These will be branded generic products. The Alpine 80 (Box) cigarettes deliver 15 mg tar, 0.9 mg nicotine and 0.7 mg smoke menthol; the Alpine Lights cigarettes deliver 9 mg tar, 0.7 mg nicotine and 0.6 mg smoke menthol.

R. J. Reynolds is test marketing More White Lights 120 (plain and menthol) cigarettes in New York City. These cigarettes have a 21 mm circumference and white cigarette paper. The More Lights currently on the market have brown paper. The plain version delivers 9 mg tar and 0.7 mg nicotine; the menthol version delivers 11 mg tar, 0.7 mg nicotine and 0.5 mg smoke menthol.

R. J. Reynolds is distributing Best Value generic cigarettes in K-Mart stores. These are the first generic products from Reynolds. The blend of these products contains a high level of reconstituted material (30%). Extruded tobacco filaments (3-4%) were incorporated in the blend of these cigarettes. The binder for the extruded tobacco is glycerin. The Full Flavor cigarettes deliver 15 mg tar and 1 mg nicotine; the Lights cigarettes deliver 11 mg tar and 0.8 mg nicotine; and the Ultra Light cigarettes deliver 6 mg tar and 0.5 mg nicotine. The smoke menthol delivery is 0.5 mg.

The Liggett Group has introduced nationally Pyramid 85 (NF), Pyramid Lights 85 and 100, Pyramid Lights 100 (Menthol) and Pyramid Ultra Lights 100 cigarettes. These sub-generic priced cigarettes will sell for $2.50 less per carton ($5.00-$7.00) than generic cigarettes. The nonfilter cigarette delivers 21 mg tar and 1.4 mg nicotine, the Lights cigarettes deliver 14 mg tar and 1.1 mg nicotine; the Ultra Lights cigarettes deliver 6 mg tar and 0.6 mg nicotine. The smoke menthol delivery is 0.5 mg.

Brown & Williamson is test marketing Kool Ultra Lights 85 and 100 and Kool Lights 85 and 100 cigarettes in St. Louis, Missouri. These cigarettes have new package graphics and white tipping. The Kool Ultra and Kool Lights currently on the

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market have cork tipping. No changes were noted in smoke deliveries between the Kool Ultra and Kool Lights currently on the market and the test market brands.

2. Brand Modifications

Reynolds has reduced the tobacco weight of Winston 85 (0.751 to 0.720 g). This lower tobacco weight was seen in Winston 85 only manufactured since October, 1988. This resulted in a lower total RTD (128 to 118 mm of H_2O) and puff count (8.7 to 8.1). A lower ventilation level (9 to 6%) and higher total alkaloids (1.87 to 2.02%) were observed. No changes were observed in smoke deliveries.

II. ANALYTICAL METHODS DEVELOPMENT AND SUPPORT

A. **Objective**: To evaluate and recommend analytical methods and new technology in support of programs for R&D and Manufacturing.

B. **Results**:

"Mt. Smoky", a cigarette holder from Japan, was microscopically examined. The holder consisted of a plastic cigarette holder and a filter plug. The two pieces appeared to be glued together. The pieces were separated and inside the one closest to the mouthend was a small filter made of cotton fibers. The cotton fibers were wrapped with a typical plugwrap paper consisting of flax, shive and hardwood fibers. Both the cotton filter and the plugwrap paper were coated with menthol crystals.

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